

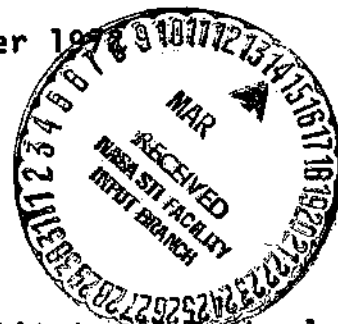
7
11/1/66

(NASA-CR-130808) DESIGN AND DEVELOPMENT
OF DIBORANE SHIPPING CONTAINER. VOLUME
2, APPENDIX A: RECORD OF CONTACTS WITH
DOT REGARDING SPECIAL PERMIT FOR Callery
Chemical Co., 94 p HC \$6.75 CSCI 13D
N73-18466
63/15 Unclass
17082

APPENDIX A
RECORD OF CONTACTS WITH D.O.T.
REGARDING SPECIAL PERMIT FOR
200-POUND DIBORANE SHIPPING CONTAINER

VOLUME II
OF
FINAL REPORT
FOR
DESIGN AND DEVELOPMENT OF DIBORANE SHIPPING CONTAINER

For Period
7 November 1968 - 18 September 1972



Prepared for
Jet Propulsion Laboratory, California Institute of Technology
Pasadena, California 91103
Under
National Aeronautics and Space Administration
Contract NASW-1827

Submitted by
Callery Chemical Company
Division of Mine Safety Appliances Company
Callery, Pennsylvania
16024

TABLE OF CONTENTS

	Page
INTRODUCTION.....	A- 1
PART 1 - PETITION TO D.O.T. FOR SPECIAL PERMIT.....	A- 3
<u>4 June 1971</u> [CCC 71-12] Design and Performance 200-pound Diborane Shipping Container.....	A- 3
Cover Letter.....	A- 4
Background.....	A- 7
Design.....	A- 9
Performance.....	A-14
Data Tables.....	A-18
Drawings.....	A-35
PART 2 - ADDENDA TO PETITION TO D.O.T. FOR SPECIAL PERMIT.....	A-36
<u>29 July 1971</u> (Quantities less than 200 pounds).....	A-37
<u>20 August 1971</u> (Qualification test shipment with methanol).....	A-40
<u>10 December 1971</u> (Shipment by railway express).....	A-42
<u>3 March 1972</u> (Quantities less than 100 pounds).....	A-44
<u>15 March 1972</u> (Rewording to prevent misinter- pretation).....	A-47
<u>28 March 1972</u> (Withdrew request for less than 100 pounds; expedite other requests).....	A-50
<u>21 April 1972</u> (Shipment of recoolled container).....	A-52
Cover Letter.....	A-52
Shipment of Recooled Containers.....	A-54
Thermal Performance-Recooling.....	A-55
Proposed Rewording, DOT S.P. 6522.....	A-75
<u>24 April 1972</u> (Recooling provisions).....	A-79
<u>30 May 1972</u> (Rewording of "empty container" definition).....	A-81
<u>30 August 1972</u> (Rewording of recooling provisions).....	A-82
<u>22 September 1972</u> (JPL request, less than 100 pounds)...	A-88

LIST OF TABLES

	Page
Table A-1	30-Day Storage Test Results Smoothed Data-Even Days.....A-17 (Table 1)
Table A-2	30-Day Storage Test Results Smoothed Data-Linear Segments.....A-20 (Table 2)
Table A-3	Precooling for 30-Day Storage Test.....A-21 (Table 3)
Table A-4	Diborane Charge for 30-Day Storage Test.....A-23 (Table 4)
Table A-5	30-Day Storage Test - Complete Data.....A-28 (Table 5)
Table A-6	Post-Test Recool.....A-33 (Table 6)
Table A-7	Diborane Shipping Container Extrapolated Test Data (80 to 201.5 pounds).....A-39
Table A-8	Diborane Shipping Container Extrapolated Test Data (5 to 201.5 pounds).....A-46
Table A-9	Diborane Shipping Container Post-Test Recool.....A-59 (Table 4)

LIST OF FIGURES

	Page
Figure A-1	Diborane Shipping Container Elevation View.....A-12 (Figure 1)
Figure A-2	Diborane Shipping Container Plan View.....A-13 (Figure 2)
Figure A-3	30-Day Storage Test Results.....A-16 (Figure 3)
Figure A-4	True Temperature Based On Indicated Pressure.....A-65 (Figure 4)
Figure A-5	Recooling Data.....A-66 (Figure 5)
Figure A-6	Storage Test Data.....A-72 (Figure 6)
Figure A-7	Predicted Thermal Performance for -70°C. Initial Temperature & 200 lbs. B ₂ H ₆ .A-73 (Figure 7)
Figure A-8	Diborane Heat Capacity Data.....A-74 (Figure 8)

INTRODUCTION

This compilation, comprising Appendix A (Volume II) of Final Report for "Design and Development of Diborane Shipping Container", consists of pertinent documents of record in contacts with Department of Transportation to obtain and revise Special Permit No. 6522.

Each document is included as sent to DOT, although in some cases additional notes have been added (and identified as such). Individual documents have page numbers at the top, while appendix page numbers (A-1, A-2, etc.) have been added at the bottom. The same situation applies to table numbers and figure numbers, which have each been given consecutive numbers A1, A2, etc. in addition to the original numbers.

These documents have been grouped in two parts. First is the initial petition to DOT, submitted in June 1971 to request a special permit. The second part contains addenda, including support of the initial petition and requests for revisions to the permit after it had been issued.

DOT Special Permit No. 6522 was received on 27 December 1971. First Revision to the permit was received on 24 April 1972. Second Revision (current) to DOT Special Permit No.

6522 was received on 18 September 1972, with an expiration date of 15 September 1972.

CCC 71-12

200-POUND DIBORANE SHIPPING CONTAINER

DESIGN & PERFORMANCE

Developed Under Contract Number NASW 1827

SUBMITTED JUNE 1971

by

CALLERY CHEMICAL COMPANY

DIVISION OF MINE SAFETY APPLIANCES COMPANY

CALLERY, PENNSYLVANIA

16024



CALLERY, PENNSYLVANIA 16024 • PHONE (412) 538-3510

14 June 1971

Office of the Secretary of Transportation
Washington, D. C. 20590

Attention: Mr. Gordon Rousseau
Chief, Special Permits Branch
Office of Hazardous Materials

Dear Mr. Rousseau:

Under Contract Number NASW 1827, Callery Chemical Company has developed and tested a new container for shipment of diborane.

The new container is designed for shipment of 200 pounds of liquid diborane; and it incorporates the same design concepts of safety and reliability which have permitted Callery to accumulate an excellent shipping record for diborane in quantities up to 40 pounds.

The attached document gives the container performance test data, along with complete as-built drawings. Safe time for shipment was shown to be over thirty (30) days; therefore, we feel that a permit based on fifteen (15) days would give a comfortable margin of safety, while expediting delivery.

Mr. Gordon Rousseau

- 2 -

Callery Chemical Company hereby requests your favorable consideration in granting a special permit for use of this container. Please advise if any further information is required for your decision on this special permit application.

Very truly yours,

CALLERY CHEMICAL COMPANY
DIVISION OF MSA COMPANY



K. W. Beahm
Project Leader

KWB:jp

Attachments

cc: Mr. R. M. Graziano, Agent
Bureau of Explosives
Association of American Railroads
1920 L. Street, N. W.
Washington, D. C. 20036

Mr. W. B. Powell, Technical Manager (2 copies)
Jet Propulsion Laboratory
California Institute of Technology
Building 125, Room 224
4800 Oak Grove Drive
Pasadena, California 91103

NASA
Headquarters Contracts Division
Washington, D. C. 20546
Attn: The Contracting Officer, Code DHC-6
Subject: Contract NASW-1827

Mr. Frank E. Compitello/Code RPL
NASA-OART
Liquid Propulsion Technology
600 Independence Avenue
Washington, D. C. 20546

**200-POUND DIBORANE SHIPPING CONTAINER
DESIGN AND PERFORMANCE**

TABLE OF CONTENTS

	PAGE
BACKGROUND.....	1
DESIGN.....	3
Figure 1 - Diborane Shipping Container, Elevation View.....	6
Figure 2 - Diborane Shipping Container, Plan View.....	7
PERFORMANCE.....	8
Figure 3 - 30-Day Storage Test Results.....	10
Table 1 - 30-Day Storage Test Results Smoothed Data - Even Days.....	11
DATA TABLES.....	12
Table 2 - 30-Day Storage Test Results Smoothed Data-Linear Segments.....	14
Table 3 - Precooling for 30-Day Storage Test.....	15
Table 4 - Diborane Charge for 30-Day Storage Test.....	17
Table 5 - 30-Day Storage Test - Complete Data.....	22
Table 6 - Post-Test Recool.....	27
DRAWINGS.....	29

200-POUND DIBORANE SHIPPING CONTAINER

BACKGROUND

Callery Chemical Company has been shipping diborane as a flammable compressed gas since 1953 and as a liquid since 1959. Callery holds Special Permit Number 930 for shipment of gaseous diborane and diborane-inert gas mixtures in non-refrigerated overpacks, under specific conditions outlined in the permit. Callery also holds Special Permit Number 970 for shipment of liquid diborane in overpacks containing dry ice for refrigeration. These permits were rewritten and reissued in their present wording in January 1968. Over the years Callery has an excellent record for safe shipment of diborane. In addition we have handled diborane with consistently excellent safety records in our laboratories and production plants.

Currently the largest single package permits shipment of up to 40 pounds of diborane in a DOT-3AA2400 cylinder surrounded by dry ice in an insulated box. Gross shipping weight of this package is about 1300 pounds, and Special Permit 970 requires delivery within ten (10) days.

Prospect of increased future demand for diborane renders this package inadequate for the projected quantities and impractical from the standpoint of handling labor.

In accordance with the anticipated increased use of diborane, NASA awarded Contract Number NASW-1827 to Callery Chemical Company for the design and development of a container for shipment of approximately 200 pounds of liquid diborane. Work began on 7 November 1968 and has continued to date under the same contract number.

Notification of this work was given to the Department of Transportation and to the Bureau of Explosives on 15 November 1968. Upon completion of the initial design, complete drawings were submitted to these agencies on 15 July 1969. Callery representatives met with Bureau of Explosives personnel on 24 September 1969, and the Bureau's recommendations were incorporated into the design. An exchange of comments on the design was made with D.O.T. in November and December 1969. Subsequently the container has been tested with the results presented herein.

200-POUND DIBORANE SHIPPING CONTAINER

DESIGN

Design concept for this container was selected to take full advantage of the success achieved in shipping diborane for over fifteen years. On this basis dry ice was chosen as the refrigerant; because this eliminates the need for control devices used in liquid nitrogen or other liquid flow-type refrigeration systems. In addition dry ice is comparatively much easier to obtain and add to a container which has been delayed in transit.

Detail design of the container was performed by CVI Corporation, subsidiary of Pennwalt Corporation, under subcontract to Callery. Full advantage was taken of Callery's experience in terms of materials of construction, valve types, etc. for diborane service. CVI contributed the expertise in cryogenic vessel design technology to achieve the necessary insulation efficiency and strength. Unlike Callery's previous diborane cylinder overpacks, the new container depends to a lesser degree upon the dry ice and to a greater extent on a much more effective insulation around the inner tank containing the diborane.

Reference to Figure 1 and Figure 2 will show the general layout of the new container; with a 36-inch diameter spherical

inner tank for the diborane, surrounded (except for the dry ice chamber) by a 48 inch diameter cylindrical shell containing perlite insulation evacuated to about ten microns absolute pressure. Over 100 pounds of dry ice may be added through a bellows-sealed neck into a chamber in direct contact with the top of the sphere. The spherical tank containing the diborane is coded for a maximum working pressure of 500 psig at -320 to +100°F., protected by a 550 psig rupture disk and 550 psig relief valve connected in series.

Instruments are provided to indicate temperature, pressure, and liquid level. A audio-visual alarm is activated by temperature in excess of -35°C., which should occur after about 20 days; this allows over ten days before reaching 0°C., still well below the point where pressure relief will occur.

In keeping with end use requirements of NASA, the liquid unloading rate was designed for a minimum of 0.2 pound per second with 100 psi pressure differential. Actual unloading rate of 0.2 pound per second was obtained with a pressure differential of only 20 psi.

The container is designed for handling by fork lift or by crane; gross shipping weight is about 2890 pounds. Envelope dimensions are 4'-4" diameter and 6'-6" height. Center of gravity is shown on Figure 1 and Figure 2.

All supports, components, and auxiliaries are designed to withstand, without damage or leakage, forces of 8 g vertically down and in all horizontal directions, and 4 g vertically up.

Storage time, volumetric loading, and pressure-temperature limitations are discussed in the Performance section following.

CVI Corporation has calculations for strength of the inner vessel support system, and these could be made available for review if necessary.



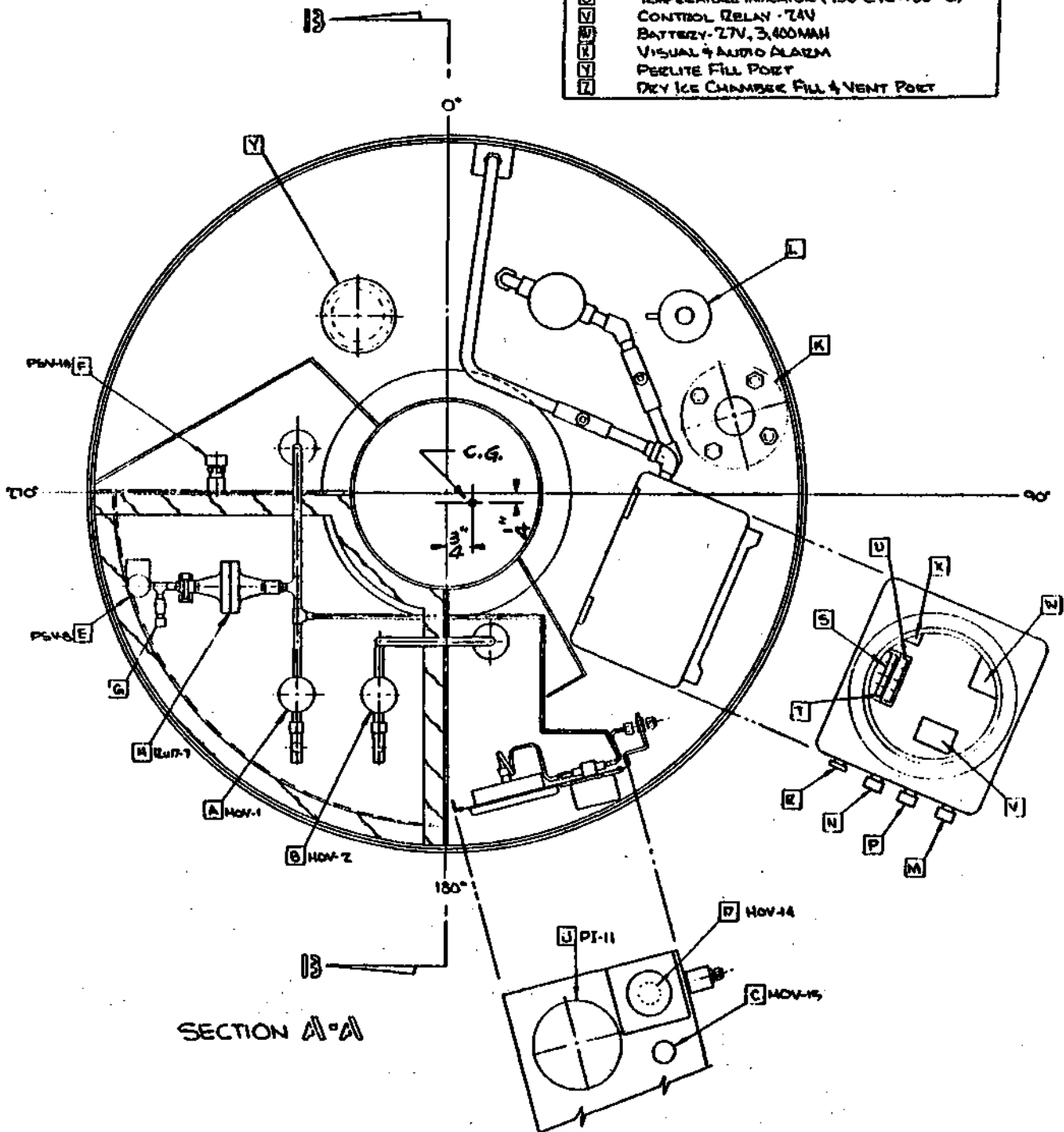
FIGURE 1 DIBORANE SHIPPING CONTAINER, ELEVATION VIEW

FIGURE 2

DIBORANE SHIPPING CONTAINER PLAN VIEW

FIGURE A-2

ITEM	DESCRIPTION
A	VENT VALVE & PRESSURIZATION VALVE
B	FILL VALVE & LIQUID UNLOADING VALVE
C	PRESSURE GAGE ISOLATION VALVE
D	PRESSURE GAGE PURGE VALVE
E	SAFETY VALVE SET @ 550 PSIG
F	SAFETY VALVE SET @ .5 PSI
G	EXCESS FLOW VALVE
H	RUPTURE DISC SET @ 550 PSIG
I	PRESS. GAGE -30" VAC -0 TO 1000 PSIG RANGE
J	FILTER ASSY W/HAUSTINGS DVGW VAC GAGE
K	LINE OPERATOR P/T PUMP OUT VALVE
L	TEST-PUSH BUTTON
M	SILENCE-PUSH BUTTON
N	LEVEL INDICATOR PUSH BUTTON
O	DRY ICE CHAMBER
P	ON-OFF SELECTOR SWITCH
Q	VOLTMETER-LINEAR SCALE
R	LIQUID LEVEL INDICATOR
S	TEMPERATURE INDICATOR (-150°C TO +130°C)
T	CONTROL RELAY - 74V
U	BATTERY - 27V, 3,600MAH
V	VISUAL & AUDIO ALARM
W	PERLITE FILL PORT
X	DRY ICE CHAMBER FILL & VENT PORT



200-POUND DIBORANE SHIPPING CONTAINERPERFORMANCE

Container performance is summarized by Figure 3 and Table 1 in this section; complete information is given in the Data Tables Section following.

Limitation on safe storage or shipment time is pressure:

<u>LIQUID TEMP., °C</u>	<u>VAPOR PRESSURE, psig</u>	<u>LIQUID FILL %</u>
0	385	76
9.8	500	88
12.2	550	98
12.5	560	100

Complete liquid fill occurs at 12.5°C., whereas maximum working pressure of 500 psig is reached near 10°C. For a practical limit, Callery has chosen a pressure of 400 psig; which, allowing for small partial pressure of non-condensables, is equivalent to about 0°C. This then becomes the temperature at which the container should be iced to recool; realizing, however, that is not an absolute limit.

Secondly, from the product purity standpoint it is desirable that the normal maximum temperature be maintained about -25 to -35°C., below which decomposition is essentially nil. Initially -35°C. had been selected as the normal maximum

operating temperature; that is, the temperature reached by a container delivered within the time required by the shipping permit.

Figure 3 and Table 1 show that the dry ice was spent in fourteen days, but it was over twenty days before the contents reached -35°C . When the test was discontinued after 29 days, the pressure was 300 psig and the temperature was -10°C . Extrapolation of temperature rise at the same rate shows that 0°C . would not be reached until over 33 days. All of these data are based on an ambient temperature of 75°F .; however, ambient temperature occasionally rose as high as 92°F .

The performance achieved with this container is adequate for consideration of a 20-day shipping permit, both in terms of safety factor to locate and reice a lost shipment and in terms of preserving product purity. It should be possible, however, to routinely obtain delivery within fifteen days, particularly in view of the fact that we currently make all shipments within ten days. Use of a fifteen day permit will have the effect of expediting delivery more so than a twenty day permit. In any case the safe shipping time is well over thirty days.

FIGURE A-3

FIGURE 3

30-DAY STORAGE TEST RESULTS

Page A-16

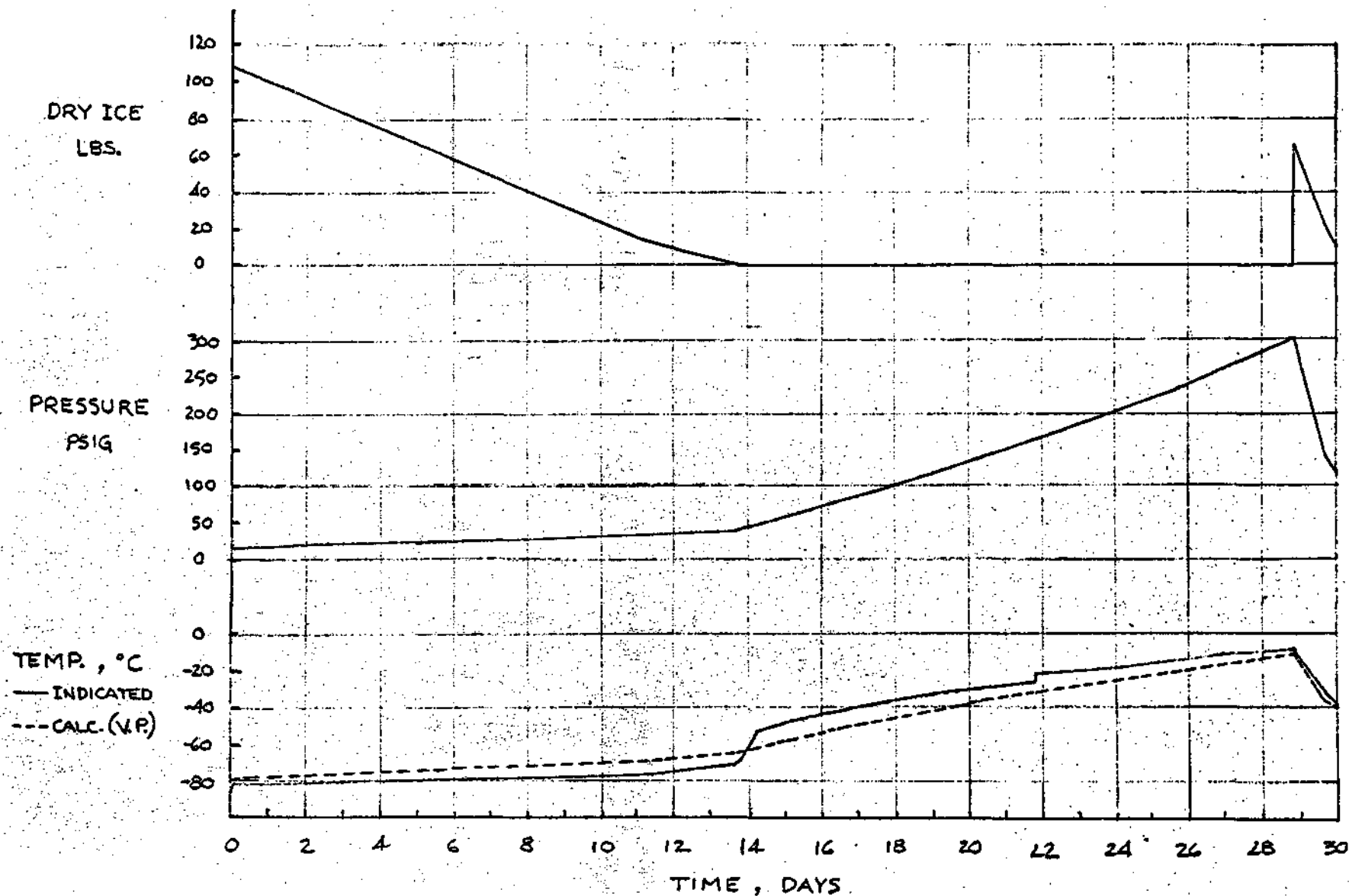


Table A-1

Table 1

DIBORANE SHIPPING CONTAINER
30-DAY STORAGE TEST RESULTS
SMOOTHED DATA-EVEN DAYS

<u>TIME, DAYS</u>	<u>DRY ICE, POUNDS</u>	<u>PRESSURE PSIG</u>	<u>TEMP., °C</u>
0	108.0	18.0	-79.0
1	101.2	21.5	-76.0
2	93.8	24.2	-74.2
3	85.4	26.5	-72.8
4	77.0	27.7	-72.1
5	68.6	29.0	-71.4
6	60.2	30.3	-70.7
7	51.8	31.6	-70.0
8	43.4	32.9	-69.3
9	35.0	34.2	-68.6
10	26.6	35.5	-67.9
11	18.2	36.8	-67.2
12	10.6	38.1	-66.6
13	4.7	40.0	-65.9
14	0	45.7	-63.5
15	0	59.5	-58.3
16	0	74.5	-53.0
17	0	89.5	-48.6
18	0	104.5	-44.3
19	0	119.5	-40.5
20	0	135.5	-37.0
21	0	151.5	-33.7
22	0	167.6	-30.7
23	0	184.6	-27.7
24	0	(192.2) 202?	-24.9
25	0	219.8	-22.1
26	0	238.6	-19.4
27	0	259.5	-16.6
28	0	280.5	-13.7
*29	0	301.5	-10.8
*30	0	322.4	- 7.9
*31	0	343.4	- 5.3
*32	0	364.4	- 2.8
*33	0	385.4	- 0.5
*34	0	406.3	+ 1.6

*Extrapolation

200-POUND DIBORANE SHIPPING CONTAINERDATA TABLES

This section contains complete data on the container preparation, charging, and storage test; as follows:

TABLE 2 30-DAY STORAGE TEST RESULTS
SMOOTHED DATA-LINEAR SEGMENTS

These results were obtained by plotting dry ice weight, container pressure, and container temperature data from Table 4; and fitting a series of straight lines through each set of data. This then became the basis for the even-day data given in Table 1.

TABLE 3 PRECOOLING for
30-DAY STORAGE TEST

Prior to charging diborane, the container was precooled by charging liquid nitrogen into the inner tank. About one week was allowed for cooling to below $-80^{\circ}\text{C}.$, and the excess liquid nitrogen was removed prior to charging with diborane.

TABLE 4 DIBORANE CHARGE for
30-DAY STORAGE TEST

Diborane was charged in two steps, separated by a topping operation to remove non-condensables (nitrogen initially present and a small quantity of inerts fed with the diborane). The initial charge of 203.5 pounds was accomplished in about four hours using an external condenser, designed and operated to add the liquid diborane at about -75°C . After topping to a net weight of 197 pounds, the second charge was made; and after retopping, the final diborane net weight was 201.5 pounds.

TABLE 5 30-DAY STORAGE TEST
COMPLETE DATA

Upon completion of the diborane charge, 108 pounds of dry ice was added; and the container was sealed for thermal testing. Ambient temperature was maintained at about 75°F .; however, with no cooling capability, this temperature occasionally ranged as high as 92°F . This phase of the test was discontinued after 29 days by adding dry ice to begin recooling, as sufficient data were available for reliable extrapolation to 0°C .

TABLE 6 POST-TEST RECOOL

Data records were continued during the recooling period to demonstrate that continued low-temperature storage is possible and practical. Temperatures below -70°C . were achieved.

Table A-2

Table 2

DIBORANE SHIPPING CONTAINER
30-DAY STORAGE TEST RESULTS
SMOOTHED DATA-LINEAR SEGMENTS

TIME DAYS	DRY ICE LBS	PRES. PSIG	TEMP °C.		REMARKS
			ACTUAL	INDIC.	
0.00	108.0	18	-79.0	-85.5	Start
0.01	-	-	-	-82	
0.10	-	19	-	-	
0.21	-	-	-77.5	-	
1.65	96.75	-	-	-	
2.65	-	26	-73.0	-	
11.29	15.75	-	-	-	
11.64	-	-	-	-77.5	
12.29	8.5	-	-	-	
12.68	-	39	-	-	
13.10	-	-	-65.8	-	
13.64	-	-	-	-71	
13.67	-	42	-65.0	-	
13.88	0	-	-	-68	
14.02	-	-	-	-61	
14.30	-	49	-62.1	-54	
15.10	-	-	-	-47	
15.81	-	-	-53.8	-	
16.66	-	-	-	-41	
17.86	-	-	-	-38.5	
18.29	-	-	-43.0	-	
19.03	-	120	-	-	
19.10	-	-	-	-32	Alarm
19.66	-	-	-	-30	
20.66	-	-	-34.7	-	
21.87	-	-	-	-26	
21.89	-	-	-	-21.5	Slosh
22.34	-	173	-	-	
23.22	-	-	-27.0	-	
24.65	-	-	-	-18	
25.64	-	231	-	-	
26.64	-	-	-17.6	-	
26.96	-	-	-	-10	
28.93	0	300	-11.0	-9	
28.94	65.5	290	-12.3	-10	Dry Ice Added
29.72	22.5	140	-36.0	-33	
29.98	13.5	125	-39.2	-39	

DIBORANE SHIPPING CONTAINER - PRECOOLING

TIME	DATE	CONTAINER			AMBIENT TEMP. °F.	SCALE READING LBS.	LN ₂ WEIGHT LBS.	REMARKS
		TI °C	PI PSIG	VACUUM MICRONS				
1320	4/13/71	29	-	24.5	-	2582.5	0	Started adding LN ₂
1326	4/13/71	29	-	7.0	-	2583.5	1	
1328	4/13/71	29	-	5.5	-	2584	1.5	
1335	4/13/71	29	-	4.0	-	2585	2.5	
1350	4/13/71	29	-	3.5	-	2589	6.5	
1410	4/13/71	26	-	3.0	-	2603	20.5	LN ₂ off LN ₂ on
1425	4/13/71	25	-	2.9	-	2608	25.5	
1450	4/13/71	21	-	6.0	-	2602	19.5	
1503	4/13/71	20	-	3.0	-	2611	28.5	
1505	4/13/71	-	-	-	-	-	-	
1540	4/13/71	19	-	2.5	-	2678	95.5	Compl 1st 50 l. Start 2nd 50 l. Compl 2nd 50 l. Start 3rd 50 l. (half full) Compl 3rd 50 l.
1542	4/13/71	-	-	-	-	-	-	
1550	4/13/71	19	-	2.3	-	2695	112.5	
1605	4/13/71	14	19	16	-	2691	108.5	
1625	4/13/71	-	-	23	-	-	-	
0810	4/14/71	-60	30	0.5	50	2648	65.5	LN ₂
0915	4/14/71	-62	30	0.8	53	2647	64.5	
1300	4/14/71	-70	30	0.9	58	2640	57.5	
1650	4/14/71	-74	29	0.6	60	2634	51.5	
2200	4/14/71	-80	29	0.1	56	2627	44.5	
0815	4/15/71	-89	30	0	50	2614	31.5	
0920	4/15/71	-88	29	0.3	55	2613	30.5	
1340	4/15/71	-88	29	0.9	65	2608	25.5	
1650	4/15/71	-88	29	0.8	63	2604	21.5	
2330	4/15/71	-88	28	0.2	54	2598	15.5	
0810	4/16/71	-90	30	0	54	2591	8.5	
0915	4/16/71	-90	8	0	56	2795	212.5	
1200	4/16/71	-92	18	1.0	67	2793	210.5	
1625	4/16/71	-93	29	1.4	69	2793	210.5	
1200	4/17/71	-100	39	0.3	60	2781	198.5	
2245	4/17/71	-101	39	0.2	59	2771	188.5	

TABLE A-3 PAGE 2 OF 2

TABLE 3 PAGE 2 OF 2

DIBORANE SHIPPING CONTAINER - PRECOOLING

TIME	DATE	CONTAINER			AMBIENT TEMP. °F.	SCALE READING LBS.	LN2 WEIGHT LBS.	REMARKS
		TI °C	PI PSIG	VACUUM MICRONS				
1320	4/18/71	-103	39	1.1	70	2754	171.5	
0830	4/19/71	-103	38	0.4	61	2732	149.5	
0905	4/19/71	-102	40	0.5	63	2731	148.5	
1545	4/19/71	-102	38	2.1	79	2723	140.5	
2200	4/19/71	-103	38	1.0	65	2716	133.5	
0810	4/20/71	-102	40	0.2	57	2705	122.5	
1115	4/20/71	-103	75	2.8	70	2704	121.5	Leak Testing Removed LN2
1345	4/20/71	-102	205	1.7	73	-	-	
1620	4/20/71	-102	5	2.0	78	2588.5	-	
2000	4/20/71	-102	5	1.7	71	2588	-	
0805	4/21/71	-100	5	0.5	60	2588	-	

DIBORANE SHIPPING CONTAINER
DIBORANE CHARGE FOR 30-DAY STORAGE TEST

TIME	DATE	CONTAINER					COND. INLET PSIG	AMB TEMP °F	SCALE READING LBS	B ₂ H ₆ WEIGHT LBS	CHARGE TIME MIN.	REMARKS
		TI °C	PI PSIG	VI* %	LI* %	VAC mm						
0805	4/21/71	-100	5	-	0	0.5	-	60	-	-	-	Gas sample 1
0905	4/21/71	-	-	-	-	-	-	-	2580	-	-	Tare disconnected
1000	4/21/71	-	-	-	-	-	-	-	2600	-	-	Tare connected
1105	4/21/71	-98	0	-	-	0.6	-	61	2617	-	-	Dry ice added
1129	4/21/71	-	0	-	-	-	110	-	-	-	-	Open to cond.
1132	4/21/71	-98	0	80	0	0.4	-	59	2617	-	-	
1144	4/21/71	-	0	-	-	-	110	-	2617	0	0	Started charge
1146	4/21/71	-	10	-	-	-	-	-	2618	1	2	
1147	4/21/71	-98	10.5	-	-	-	-	-	2620	3	3	
1148	4/21/71	-	10.5	-	-	-	-	-	2622	5	4	
1150	4/21/71	-	10.5	-	-	-	101	-	2624	7	6	
1151	4/21/71	-	11	-	-	-	-	-	2625	8	7	
1153	4/21/71	-	11	-	-	-	96	-	2625	8	9	Dip tube plug
1156	4/21/71	-	11	-	-	-	82	-	2627	10	12	Tube opened
1157	4/21/71	-	11	-	-	-	84	-	2628	11	13	
1157.5	4/21/71	-98	12	-	-	-	84	-	2630	13	13.5	
1159	4/21/71	-98	13	80	7	-	90	-	2633	16	15	
1200	4/21/71	-98	-	-	-	-	-	-	2634	17	16	Closed B ₂ feed
1233	4/21/71	-97	-	-	-	-	126	57	2635	18	-	
1241	4/21/71	-	14	-	-	-	122	-	2635	18	16	Opened B ₂ feed
1242	4/21/71	-	-	-	-	-	-	-	2636	19	17	
1243	4/21/71	-	14	-	-	0.4	-	-	2638	21	18	
1243.5	4/21/71	-	-	-	-	-	-	-	2639	22	18.5	Reduced feed rate
1244	4/21/71	-	14	-	-	-	-	-	2641	24	19	
1245	4/21/71	-	16	-	-	-	-	-	2642	25	20	
1246	4/21/71	-	-	-	-	-	-	-	2644	27	21	
1248	4/21/71	-97	19	-	15	-	-	-	2646	29	23	
1249	4/21/71	-	-	-	-	-	-	-	2648	31	24	Reduced feed rate
1250	4/21/71	-	-	-	-	-	104	-	2649	32	25	
1251	4/21/71	-	20	-	-	-	-	-	2650.5	33.5	26	
1253	4/21/71	-	20	-	-	-	-	-	2652.5	35	28	
1255	4/21/71	-	-	-	-	-	-	-	2653	36	30	
1257.5	4/21/71	-	-	-	-	-	-	-	2655	38	32.5	

*VI = Voltage Indicator; LI = Level Indicator [Footnote added October 1972]

DIBORANE SHIPPING CONTAINER
DIBORANE CHARGE FOR 30-DAY STORAGE TEST

TIME	DATE	CONTAINER					COND INLET PSIG	AMB TEMP °F	SCALE READING LBS	B ₂ H ₆ WEIGHT LBS	CHARGE TIME MIN.	REMARKS
		TI °C	PI PSIG	VI %	LI %	VAC						
1259	4/21/71	-	20	-	-	-	-	-	2657	40	34	
1301	4/21/71	-	-	-	-	-	98	-	2659	42	36	
1304	4/21/71	-96	20	-	21	-	-	-	2661	44	39	
1306	4/21/71	-	20	-	-	-	-	-	2663	46	41	
1308	4/21/71	-	21	-	-	-	-	-	2665	48	43	
1311	4/21/71	-	21	-	-	-	94	-	2667	50	46	
1313	4/21/71	-96	21	-	-	-	94	-	2669	52	48	
1316	4/21/71	-	22	-	-	-	-	-	2671	54	51	
1318	4/21/71	-	24	-	-	-	-	-	2673	56	53	
1321	4/21/71	-95	24	-	-	-	90	-	2675	58	56	
1323	4/21/71	-	-	-	-	-	-	-	2677	60	58	
1325	4/21/71	-	25	-	-	-	87	-	2679	62	60	
1328	4/21/71	-	27	-	-	-	-	-	2681	64	63	
1331	4/21/71	-	27	-	-	-	86	-	2683	66	66	
1333	4/21/71	-	-	-	-	-	-	-	2685	68	68	
1336	4/21/71	-	30	-	30	-	-	-	2687	70	71	
1338	4/21/71	-95	30	-	-	-	84	-	2689	72	73	
1340.5	4/21/71	-	30	-	-	-	-	-	2691	74	75.5	
1345	4/21/71	-	30	-	-	-	-	-	2695	78	80	
1348	4/21/71	-	30	-	-	-	-	-	2697	80	83	
1350.5	4/21/71	-	-	-	-	-	87	-	2699	82	85.5	
1353	4/21/71	-	-	-	-	-	-	-	2701	84	88	
1355	4/21/71	-	-	-	-	-	-	-	2703	86	90	
1358	4/21/71	-95	31	-	35	-	92	-	2705	88	93	
1359.5	4/21/71	-	31	-	-	-	-	-	2707	90	94.5	
1401	4/21/71	-	31	-	-	-	-	-	2709	92	96	
1403.5	4/21/71	-	-	-	-	-	93	-	2711	94	98.5	
1406	4/21/71	-	32	-	-	-	-	-	2713	96	101	
1408.5	4/21/71	-	-	-	-	-	-	-	2715	98	103.5	
1411	4/21/71	-	35	-	-	-	93	-	2717	100	106	
1413	4/21/71	-95	35	-	38	-	-	-	2719	102	108	
1415	4/21/71	-95	35	-	-	-	93	-	2721	104	110	

DIBORANE SHIPPING CONTAINER
DIBORANE CHARGE FOR 30-DAY STORAGE TEST

TIME	DATE	CONTAINER					COND INLET PSIG	AMB TEMP °F	SCALE READING LBS	B ₂ H ₆ WEIGHT LBS	CHARGE TIME MIN.	REMARKS
		TI °C	PI PSIG	VI %	LI %	VAC M						
1417	4/21/71	-	37	-	-	-	-	-	2723	106	112	
1419	4/21/71	-	37	-	-	-	95	-	2725	108	114	
1421	4/21/71	-	38	-	-	0.2	-	55	2727	110	116	
1423	4/21/71	-	39	-	-	-	-	-	2729	112	118	
1426	4/21/71	-	39	-	-	-	98	-	2731	114	121	
1428	4/21/71	-	40	-	43	-	-	-	2733	116	123	
1430	4/21/71	-	40	-	-	-	-	-	2735	118	125	
1431.5	4/21/71	-	40	-	-	-	100	-	2737	120	126.5	
1433.5	4/21/71	-	40	-	-	-	-	-	2739	122	128.5	
1436	4/21/71	-	40	-	-	-	98	-	2741	124	131	
1438	4/21/71	-	40	-	-	-	-	-	2743	126	133	
1440	4/21/71	-	40	-	-	-	-	-	2745	128	135	
1443	4/21/71	-	40	-	-	-	-	-	2747	130	138	
1445	4/21/71	-	40	-	-	-	-	-	2749	132	140	
1506	4/21/71	-91	43	-	51	0.2	-	55	2767.5	150.5	161	
1509	4/21/71	-	43	-	-	-	-	-	2770	153	164	
1515	4/21/71	-91	45	-	-	-	82	-	2775	158	170	
1522	4/21/71	-91	46	-	-	-	81	-	2780	163	176	
1528	4/21/71	-91	47	-	-	-	78	-	2785	168	182	
1535	4/21/71	-91	48	-	-	-	76	-	2790	173	189	
1540	4/21/71	-91	49	-	60	-	75	-	2793	176	194	
1544	4/21/71	-91	49	-	-	-	72	-	2796	179	198	
1550	4/21/71	-	50	-	-	-	71	-	2798.5	181.5	204	
1552	4/21/71	-	50	-	-	-	-	-	2800	183	206	
1554	4/21/71	-91	50	-	-	0.2	69	56	2801	184	208	
1558	4/21/71	-91	50	-	-	-	68	-	2803	186	212	
1601	4/21/71	-91	50	-	-	-	65	-	2805	188	215	
1603	4/21/71	-	50	-	-	-	-	-	2806	189	217	
1606	4/21/71	-90.5	50	-	-	-	64	-	2807	190	220	
1609	4/21/71	-90.5	50	-	-	-	-	-	2809	192	223	
1612	4/21/71	-90	50	-	-	-	64	-	2810	193	226	
1618	4/21/71	-90.5	50	-	-	-	62	-	2812	195	232	

TABLE A-4 PAGE 4 OF 5

DIBORANE SHIPPING CONTAINER
DIBORANE CHARGE FOR 30-DAY STORAGE TEST

TIME	DATE	TI °C	PI PSIG	VI %	LI %	VAC	COND INLET PSIG	AMB TEMP °F	SCALE READING LBS	B ₂ H ₆ WEIGHT LBS	CHARGE TIME MIN.	REMARKS
1620	4/21/71	-90/5	50	-	65	-	62	-	2813.5	196.5	234	
1624	4/21/71	-90.5	50	-	-	-	63	-	2815	198	238	
1627	4/21/71	-90	50	-	-	-	63	59	2816	199	241	
1632	4/21/71	-90	50	-	-	-	62	-	2816.5	199.5	246	
1636	4/21/71	-90	50	-	65	0.2	62	58	2817	200	250	
1643	4/21/71	-90	50	-	-	-	60	57	2818	201	257	
1646	4/21/71	-90	51	79	-	-	60	-	2818	201	260	
1650	4/21/71	-90	51	-	65	0.2	58	57	2818.5	201.5*	264	Closed B ₂ feed.
1920	4/21/71	-89.5	55	78	65	0.1	-	52	2818	-	-	
2340	4/21/71	-89	55	78	65	0	-	48	2817	-	-	
0825	4/22/71	-87	58	78	65	0	-	50	2815	-	-	
0835	4/22/71	-	-	-	-	-	-	-	2803.5	203.5*	-	Weight w/o dry ice
1115	4/22/71	-86	29	-	-	0.3	-	49	2809.5	-	-	Started topping @0845
1200	4/22/71	-86	26	-	-	0.3	-	49	2808	-	-	Stopped topping
1242	4/22/71	-	22	-	-	-	-	-	2811	-	-	Started topping
1310	4/22/71	-86	22	-	-	0.3	-	49	2807	-	-	
1330	4/22/71	-	20	-	-	-	-	-	2806.5	-	-	
1345	4/22/71	-86	19	-	-	-	-	55	2805.5	-	-	Gas Sample 2
1350	4/22/71	-86	18	-	-	-	-	-	2805.5	-	-	Finished topping
1400	4/22/71	-	-	-	-	-	-	-	2796.5	196.5	-	Weight w/o dry ice
1403	4/22/71	-86	18	-	-	-	-	-	2805	197	264	{ Weight with dry ice Restarted B ₂ feed
1405	4/22/71	-	19	-	-	-	105	-	2805.5	197.5	266	
1406	4/22/71	-	20	-	-	-	-	-	2806	198	267	
1407	4/22/71	-	-	-	-	-	-	-	2806	198	268	Increased feed rate
1408	4/22/71	-	20	-	-	-	-	-	2807	199	269	
1409	4/22/71	-	20	-	-	-	-	-	2808	200	270	
1410	4/22/71	-	-	-	-	0.3	90	49	2809	201	271	
1411	4/22/71	-	21	-	-	-	-	-	2810	202	272	
1411.5	4/22/71	-	21	-	-	-	-	-	2810.5	202.5	272.5	{ Stopped feeding Weight with dry ice Weight w/o dry ice
1412	4/22/71	-	-	-	-	-	-	-	2811	203	273	
1415	4/22/71	-	-	-	-	0.2	110	50	2803	203.0	-	
1525	4/22/71	-86	18	-	-	0.3	-	51	2810.75	203.0	-	Weight with dry ice

*Actual B₂H₆ weight 203.5 due to 2.0 lbs. dry ice loss during charging.

TABLE A-4 PAGE 5 OF 5
TABLE 4 PAGE 5 OF 5

DIBORANE SHIPPING CONTAINER
DIBORANE CHARGE FOR 30-DAY STORAGE TEST

TIME	DATE	TI °C	PI PSIG	VI %	LI %	VAC %	COND INLET PSIG	AMB TEMP °F	SCALE READING LBS	B ₂ H ₆ WEIGHT LBS	CHARGE TIME MIN.	REMARKS
1540	4/22/71	-	-	78	66	-	-	-	2810.5	203.5	-	Start topping
1545	4/22/71	-86	19	-	-	0.3	-	52	2810	-	-	
1555	4/22/71	-86	18	-	-	-	-	-	2810	-	-	{ Gas Sample 3
1600	4/22/71	-86.5	18	78	65	0.3	-	53	2809.5	-	-	{ Finished topping
1601	4/22/71	-	-	-	-	-	-	-	2801.5	201.5	-	Weight w/o dry ice
1615	4/22/71	-	-	-	-	-	-	-	2781.5	201.5	-	Disconnected
1617	4/22/71	-	-	-	-	-	-	-	2783	-	-	PSV Connected
1645	4/22/71	-85.5	18	78	0	0.3	-	54	2891	-	-	{ Dry ice charged Start of test

NOTE: In the original publication to DOT, this page of this table had incorrectly been labeled "TABLE 5". Correction made October 1972.

TABLE A-5 PAGE 1 OF 5
TABLE 5 PAGE 1 OF 5

DIBORANE SHIPPING CONTAINER
30-DAY STORAGE TEST

TIME	DATE	DAYS	CONTAINER					AMB TEMP °F	SCALE READING LBS	DRY ICE, LBS.	REMARKS
			TI °C	PI PSIG	VI * %	LI * %	VACUUM MICRONS				
1645	4/22/71	0.00	-85.5	18	78	0	0.3	-	2891.0	108	Start of test
1700	4/22/71	0.01	-82	18	-	-	0.9	74	2891.0	108	
1915	4/22/71	0.10	-82	19	-	-	1.4	69	2890.75	107.75	
2145	4/22/71	0.21	-82	19.5	-	-	1.8	73	2890.0	107	
2400	4/22/71	0.30	-82	19.5	-	-	1.8	71	2889.25	106.25	
0810	4/23/71	0.64	-82	20.5	79	0	2.0	72	2887.0	104	
0840	4/23/71	0.66	-82	20.5	-	-	2.0	71	2887.0	104	
1505	4/23/71	0.93	-82	21	-	-	2.0	72	2885.0	102	
1645	4/23/71	1.00	-82	21	81	0	2.1	76	2884.5	101.5	
1915	4/23/71	1.10	-82	22	-	-	2.3	77	2883.75	100.75	
0045	4/24/71	1.33	-82	23	82	0	2.4	78	2881.75	98.75	
0815	4/24/71	1.65	-82	23	82	0	2.3	74	2879.75	96.75	
1755	4/24/71	2.05	-82	24	82	0	2.3	72	2876.25	93.25	
0815	4/25/71	2.65	-82	26	82	0	2.3	73	2872.0	89	
1330	4/25/71	2.86	-82	26	82	0	2.6	74	2870.0	87	
2400	4/25/71	3.30	-82	27	82	0	2.7	77	2866.0	83	
0815	4/26/71	3.65	-82	27.5	81	0	2.5	82	2863.5	80.5	
0845	4/26/71	3.67	-82	27.5	82	0	2.7	73	2863.25	80.25	
1525	4/26/71	3.94	-82	28	-	-	2.7	72	2861.5	78.5	
2345	4/26/71	4.29	-81	28	82	0	2.9	74	2857.75	74.75	
0810	4/27/71	4.64	-81	29	82	0	2.8	74	2855.0	72	
0840	4/27/71	4.66	-81	29	82	65	2.9	73	2854.5	71.5	
1305	4/27/71	4.85	-81	29	82	65	3.0	72	2852.75	69.75	
1600	4/27/71	4.97	-81	29	-	-	3.0	76	2852.0	69	
1645	4/27/71	5.00	-80.5	29.5	81	65	2.9	73	2851.75	68.75	
1930	4/27/71	5.11	-80.5	29.5	81	65	3.1	77	2850.75	67.75	
2400	4/27/71	5.30	-80.5	29.5	81	65	3.3	75	2849.0	66.0	
0810	4/28/71	5.64	-80.5	30	81	65	3.0	72	2846.0	63.0	
0845	4/28/71	5.67	-80	30	81	65	3.2	74	2846.0	63.0	
1500	4/28/71	5.93	-80	30.5	81	65	3.8	77	2844.0	61.0	
1930	4/28/71	6.11	-80	31	81	65	3.4	75	2842.0	59.0	

*VI = Voltage Indicator; LI = Level Indicator [Footnote added October 1972]

TABLE A-5 PAGE 2 OF 5
TABLE 5 PAGE 2 OF 5

DIBORANE SHIPPING CONTAINER
30-DAY STORAGE TEST

TIME	DATE	DAYS	CONTAINER				VACUUM MICRONS	AMB TEMP °F	SCALE READING LBS	DRY ICE, LBS.	REMARKS
			TI °C	PI PSIG	VI %	LI %					
2345	4/28/71	6.29	-80	31	81	65	3.4	74	2840.75	57.75	
0810	4/29/71	6.64	-80	31	81	65	3.5	76	2837.5	54.5	
0845	4/29/71	6.67	-80	31	81	0	3.7	75	2837.75	54.75	
1320	4/29/71	6.86	-79.5	31	82	0	3.7	76	2835.75	52.75	
1500	4/29/71	6.93	-80	31	-	-	3.5	78	2835.5	52.5	
1615	4/29/71	6.98	-79	32	82	0	3.8	78	2835.0	52.0	
1915	4/29/71	7.10	-79	32	82	0	3.7	75	2834.0	51.0	
2350	4/29/71	7.30	-79	32	82	0	3.7	75	2832.25	49.25	
0810	4/30/71	7.64	-79	32	81	65	3.8	74	2829.5	46.5	
0840	4/30/71	7.66	-79	33	81	65	3.9	75	2829.25	46.25	
1555	4/30/71	7.97	-79	32	-	-	3.8	72	2827	44.0	
1600	4/30/71	7.97	-79	33	81	65	4.0	77	2826.75	43.75	
1915	4/30/71	8.10	-79	33	81	65	3.8	74	2825.75	42.75	
2345	4/30/71	8.29	-79	33.5	81	65	3.8	73	2824	41.0	
0910	5/1/71	8.68	-78.5	34	81	65	4.0	76	2821	38.0	
1745	5/1/71	9.04	-78.5	34.5	81	65	3.9	74	2818	35.0	
0820	5/2/71	9.65	-78	35	81	65	4.0	75	2812.75	29.75	
1325	5/2/71	9.86	-78	35	81	65	4.0	74	2811.0	28.0	
1915	5/2/71	10.10	-78	35	81	65	3.9	73	2808.75	25.75	
2345	5/2/71	10.29	-78	35	81	65	4.0	74	2807.25	24.25	
0810	5/3/71	10.64	-78	35	81	65	4.0	75	2804.0	21.0	
0845	5/3/71	10.67	-78	36	81	65	4.2	77	2804.0	21.0	
1550	5/3/71	10.96	-78	36	-	-	4.0	78	2801.5	18.5	
1645	5/3/71	11.00	-77.5	36	81	65	4.1	75	2801.25	18.25	
1930	5/3/71	11.11	-77.5	36	81	65	3.9	75	2800.25	17.25	
2345	5/3/71	11.29	-77.5	36	81	65	3.9	76	2798.75	15.75	
0810	5/4/71	11.64	-77.5	37	81	65	3.9	71	2796.0	13.0	
0900	5/4/71	11.68	-77.5	37	81	65	4.2	75	2796.0	13.0	

TABLE A-5 PAGE 3 OF 5

TABLE 5 PAGE 3 OF 5

DIBORANE SHIPPING CONTAINER
30-DAY STORAGE TEST

TIME	DATE	DAYS	TI °C	PI PSIG	VI %	LI %	VACUUM MICRONS	AMB. TEMP °F	SCALE READING LBS	DRY ICE, LBS.	REMARKS
1340	5/4/71	11.92	-77	37	81	65	4.2	73	2794.5	11.5	
1520	5/4/71	11.94	-77	38	-	-	4.0	72	2794	11.0	
1915	5/4/71	12.10	-77	37	81	65	4.2	78	2793	10.0	
2345	5/4/71	12.29	-76	38	81	65	4.3	75	2791.5	8.5	
0810	5/5/71	12.64	-76	39	81	65	4.2	72	2789.0	6.0	
0900	5/5/71	12.68	-75	39	81	66	4.4	74	2788.0	5.0	
1420	5/5/71	12.90	-74.5	39.5	80	67	5.1	78	2787.5	4.5	
1525	5/5/71	12.94	-75	40	-	-	4.8	75	2787.0	4.0	
1700	5/5/71	13.01	-73.5	40	81	67	4.8	75	2786.75	3.75	
1915	5/5/71	13.10	-73.5	40	81	67	4.8	78	2786	3.0	
2345	5/5/71	13.29	-72.5	41	81	67	4.8	75	2785	2.0	
0810	5/6/71	13.64	-71	42	81	67	4.9	76	2783	0	
0855	5/6/71	13.67	-71	42	81	67	5.0	78	2783.25	0.25	
1350	5/6/71	13.88	-68	43	80	67.5	5.1	74	2782.25	-0.25	
1545	5/6/71	13.96	-66	43	-	-	4.9	72	2782.5	-0.5	
1710	5/6/71	14.02	-61	46	81	68.5	5.1	75	2782.25	-0.75	
1915	5/6/71	14.10	-58.5	47	81	69	5.1	75	2782.5	-0.5	
2400	5/6/71	14.30	-54	49	81	69	5.6	75	2782.25	-0.75	
0810	5/7/71	14.64	-51	52	81	71	5.5	74	2782.25	-0.75	
0840	5/7/71	14.66	-50	53.5	81	71	5.7	75	2782.5	-0.5	
1305	5/7/71	14.85	-49.5	58	81	71	6.1	76	2782.5	-0.5	
1530	5/7/71	14.95	-49	59	-	-	6.2	78	2782.25	-0.75	
1645	5/7/71	15.00	-48	60	81	72	6.7	79	2782.5	-0.5	
1915	5/7/71	15.10	-47	61	81	72	6.6	76	2782.5	-0.5	
2345	5/7/71	15.29	-46.5	63	81	71	6.3	75	2782.5	-0.5	
1210	5/8/71	15.81	-44	72	81	71	6.6	74	2782.75	-0.25	
1755	5/8/71	16.05	-43	74	81	70	6.7	75	2782.5	-0.5	
0830	5/9/71	16.66	-41	83	81	70	6.8	74	2782.5	-0.5	
1340	5/9/71	16.87	-40.5	88	81	71	7.7	78	2782.25	-0.75	
1745	5/9/71	17.04	-40	90	81	72	8.3	78	2782.5	-0.5	

TABLE A-5 PAGE 4 OF 5

TABLE 5 PAGE 4 OF 5

DIBORANE SHIPPING CONTAINER
30-DAY STORAGE TEST

TIME	DATE	DAYS	TI °C	PI PSIG	VI %	LI %	VACUUM MICRONS	AMB TEMP °F	SCALE READING LBS	DRY ICE, LBS.	REMARKS
2030	5/9/71	17.16	-39.5	91	81	71	8.1	76	2782.5	-0.5	
2400	5/9/71	17.30	-39.5	93	81	71	7.9	76	2782.25	-0.75	
0815	5/10/71	17.65	-39.5	100	81	71	7.5	82	2782.25	-0.75	
0820	5/10/71	17.65	-39	100	-	-	7.5	-	-	-	
1320	5/10/71	17.86	-38.5	102	81	72	8.8	80	2782.5	-0.5	
1550	5/10/71	17.96	-39	102	-	-	9.0	80	2782.25	-0.75	
1600	5/10/71	17.97	-37.5	103	81	71	9.4	81	2782.5	-0.5	
1915	5/10/71	18.10	-37	106	81	71.5	9.7	80	2782.5	-0.5	
2145	5/10/71	18.21	-36	108	81	71	9.1	75	2782.5	-0.5	
2345	5/10/71	18.29	-36	109	81	71	9.1	75	2782.5	-0.5	
0810	5/11/71	18.64	-35	112	81	71	8.5	72	2782.25	-0.75	
0840	5/11/71	18.66	-35	113	81	70	8.8	76	2782.25	-0.5	
1105	5/11/71	18.76	-35	117	81	71	9.6	80	2782.5	-0.5	
1320	5/11/71	18.86	-34.5	118	81	71	10.1	81	2782.5	-0.5	
1500	5/11/71	18.93	-34	119	81	72	11.0	82	2782.5	-0.5	
1535	5/11/71	18.95	-34	119	-	-	10.5	83	2782.25	-0.75	
1645	5/11/71	19.00	-33.5	120	81	71	11.5	84	2782.5	-0.5	
1725	5/11/71	19.03	-33	120	81	71	11.5	84	2782.5	-0.5	
1915	5/11/71	19.10	-32	121	63	86	11.5	81	2782.5	-0.5	Alarm Sounded
0100	5/12/71	19.34	-31	127	55	100	10.5	76	2782.5	-0.5	
0815	5/12/71	19.65	-30.5	130	21	30	10.0	74	2782.5	-0.5	
0830	5/12/71	19.66	-30	131	55	100	10.2	75	2782.5	-0.5	
1315	5/12/71	19.85	-30	133	13	0	11.0	79	2782.5	-0.5	
1600	5/12/71	19.97	-30	136	-	-	10.5	74	2782.5	-0.5	
1715	5/12/71	20.02	-29.5	137	22	28	10.5	74	2782.5	-0.5	
1915	5/12/71	20.10	-29.5	138	24	29	10.2	75	2782.5	-0.5	
2345	5/12/71	20.29	-29.5	140	23	32	10.2	74	2782.5	-0.5	
0810	5/13/71	20.64	-29	148	24	40	9.9	72	2782.5	-0.5	
0835	5/13/71	20.66	-29	147	24	32	10.2	75	2782.5	-0.5	
1645	5/13/71	21.00	-28	152	25	30	9.8	73	2782.5	-0.5	

TABLE A-5 PAGE 5 OF 5

TABLE 5 PAGE 5 OF 5

DIBORANE SHIPPING CONTAINER
30-DAY STORAGE TEST

TIME	DATE	DAYS	TI °C	PI PSIG	VI %	LI %	VACUUM MICRONS	AMB. TEMP °F	SCALE READING LBS	DRY ICE, LBS.	REMARKS
1905	5/13/71	21.10	-28	153	21	28	9.8	75	2782.5	-0.5	
0140	5/14/71	21.37	-27.5	158	25	32	9.8	74	2782.5	-0.5	
0810	5/14/71	21.64	-26.5	160	24	39	9.8	72	2782.5	-0.5	
0845	5/14/71	21.67	-26.5	163	-	-	9.7	74	2782.5	-0.5	
1340	5/14/71	21.87	-26	166	-	-	10.5	77	2782.5	-0.5	Start Sloshing
1405	5/14/71	21.89	-21.5	166	-	-	11.0	77	2782.0	-1.0	Finish Sloshing
1915	5/14/71	22.10	-21.5	170	-	-	11.5	76	2782.5	-0.5	
0100	5/15/71	22.34	-21.5	173	-	-	11.0	80	2782.5	-0.5	
0945	5/15/71	22.71	-21.0	180	-	-	10.5	75	2782.5	-0.5	
1745	5/15/71	23.04	-20.5	187	-	-	13.0	82	2782.75	-0.25	
2200	5/15/71	23.22	-20.0	190	-	-	13.0	79	2782.75	-0.25	
0830	5/16/71	23.66	-19.5	197	-	-	12.5	75	2782.75	-0.25	
1400	5/16/71	23.89	-19.5	202	-	-	15.0	84	2782.75	-0.25	
0815	5/17/71	24.65	-18	215	25	30	12.5	72	2782.5	-0.5	
1600	5/17/71	24.97	-17	220	-	-	16	84	2782.5	-0.5	
1730	5/17/71	25.03	-18	220	-	-	17	83	-	-	
0800	5/18/71	25.64	-14	231	-	-	13	70	-	-	
0810	5/18/71	25.64	-15	232	24	30	14	72	2782.5	-0.5	
1550	5/18/71	25.96	-14	240	-	-	19	92	2782.5	-0.5	
0800	5/19/71	26.64	-11	251	-	-	16	74	-	-	
0807	5/19/71	26.64	-10.5	250	0	0	16	74	2782.5	-0.5	
1545	5/19/71	26.96	-10	260	-	-	21	91	2782.5	-0.5	
0805	5/20/71	27.64	-10	270	3	0	19	76	2782.5	-0.5	
1545	5/20/71	27.96	-9.9	280	-	-	21	85	2782.5	-0.5	
0807	5/21/71	28.64	-9.5	290	3	0	17	72	2782.5	-0.5	
0815	5/21/71	28.65	-8	290	-	-	17.5	75	-	-	
1500	5/21/71	28.93	-9	300	-	-	20	82	2782.5	-0.5	End of Test
1520	5/21/71	28.94	-10	290	-	-	19.5	80	2848.0	65.5	Dry Ice Added

DIBORANE SHIPPING CONTAINER
POST-TEST RECOOL

TIME	DATE	DAYS	TI °C	PI PSIG	VI* %	LI* %	VACUUM MICRONS	AMB. TEMP °F	SCALE READING LBS	DRY ICE, LBS.	REMARKS
0807	5/21/71	28.64	- 9.5	290	3	0	17	72	2782.5	-0.5	
0815	5/21/71	28.65	- 8	290	-	-	17.5	75	-	-	
1500	5/21/71	28.93	- 9	300	-	-	20	82	2782.5	-0.5	End of Test
1520	5/21/71	28.94	-10	290	-	-	19.5	80	2848.0	65.5	Dry Ice Added
1000	5/22/71	29.72	-33	140	-	-	10.5	70	2805.0	22.5	
1620	5/22/71	29.98	-39	125	-	-	10.0	74	2796.0	13.5	
1630	5/22/71	29.99	-41	121	-	-	9.5	74	2866.0	83.5	Dry Ice Added
2245	5/23/71	31.25	-53	70	-	-	6.0	68	2830.5	48.0	
0815	5/24/71	31.65	-59	60	5	0	4.9	68	2822.5	40.0	
1600	5/24/71	31.97	-60	57	-	-	5.9	80	2816.5	34.0	
0805	5/25/71	32.64	-62	50	5	0	5.3	76	2805.0	22.5	
1550	5/25/71	32.96	-65	48	-	-	5.5	78	2800.0	17.5	
0010	5/26/71	33.31	-63.5	43	-	-	5.0	74	2796.0	13.5	
0807	5/26/71	33.64	-66	42	5	0	4.1	69	2792.5	10.0	
1500	5/26/71	33.93	-65	40	-	-	4.3	68	2790.0	7.5	
2200	5/26/71	34.22	-66	42	-	-	4.3	71	2787.5	5.0	
0810	5/27/71	34.64	-66	42	5	0	3.9	66	2785	2.5	
1535	5/27/71	34.95	-66	45	-	-	4.5	71	2783.5	1.0	
0930	5/28/71	35.70	-55	52	-	-	4.0	63	2782.5	0	
1500	5/28/71	35.93	-52	59	-	-	5.2	72	2782.5	0	
1545	5/28/71	35.96	-60	55	-	-	4.8	68	2867.5	85.0	Dry Ice Added
2215	5/28/71	36.24	-63	47	-	-	3.4	67	2860.5	78.0	
1030	5/29/71	36.74	-68	42	-	-	2.6	65	2852.5	70.0	
1750	5/30/71	38.04	-71	37	-	-	3.8	76	2837.0	54.5	
1140	5/31/71	38.79	-72	36	-	-	4.2	74	2829.0	46.5	
2400	5/31/71	39.30	-72	34	-	-	3.8	75	2824.0	41.5	
0810	6/1/71	39.64	-72	35	5	0	3.6	72	2820.5	38	
1555	6/1/71	39.97	-72	35	-	-	5.5	88	2817.0	34.5	
2050	6/1/71	40.17	-72	32	-	-	5.4	83	2815.0	32.5	
0815	6/2/71	40.65	-72	32	5	0	4.9	78	2810.0	27.5	
1600	6/2/71	40.97	-72	31	-	-	4.9	77	2807.5	25.0	
0810	6/3/71	41.64	-72	31	5	0	4.9	77	2801.5	19.0	

*VI = Voltage Indicator; LI = Level Indicator [Footnote added October 1972]

TABLE A6 PAGE 2 OF 2

TABLE 6 PAGE 2 OF 2

DIBORANE SHIPPING CONTAINER
POST-TEST RECOOL

TIME	DATE	DAYS	TI °C	PI PSIG	VI %	LI %	VACUUM MICRONS	AMB. TEMP °F	SCALE READING LBS	DRY ICE, LBS.	REMARKS
1600	6/3/71	41.97	-72	31	-	-	6.0	88	2798.5	16	
0810	6/4/71	42.64	-72	31	-	-	4.9	76	2792.5	10	
1405	6/4/71	42.89	-72	31	-	-	7.0	90	2790.5	8	
1425	6/4/71	42.90	-72	31	-	-	6.9	86	2853.5	71	Dry Ice Added
1925	6/4/71	43.11	-73	33	-	-	5.4	83	2850	67.5	
1145	6/5/71	43.79	-74	31	-	-	4.4	80	2842	59.5	
1430	6/6/71	44.91	-74	31	-	-	4.7	83	2833	50.5	
2345	6/6/71	45.29	-74	30	-	-	3.2	70	2826.5	44	

200-POUND DIBORANE SHIPPING CONTAINER

DRAWINGS

Following is a complete list of as-built drawings for fabrication and assembly of the 200-pound diborane shipping container. A copy of each drawing is enclosed with this document.

A458-5800	Flow Schematic
A458-5801	Final Assembly
A458-5802 Sh 1 & 2	Piping and Instruments
A458-5803 Sh 1 & 2	Main Assembly
A458-5804 Sh 1 & 2	Inner Vessel - Assembly Of
A458-5805	Inner Vessel - Details
A458-5806 Sh 1 & 2	Top Head - Assembly Of
A458-5807	Top Head - Details
A458-5808	Vacuum Filter - Assembly Of
A458-5809	Bottom Head - Assembly & Detail
A458-5810	Thermal Plug - Assembly Of
A458-5811	Suspension Bracket - Assembly Of
A458-5812 Sh 1 & 2	Suspension Bracket - Details
A458-5813	Bracket-Pressure Gage
A458-5814	Standoff Ring - Assembly Of
A458-5815	Lifting Bracket Details
A458-5816	Outer Shell - Assembly Of
A458-5817	Top Panel - Assembly Of
A458-5818 Sh 1 & 2	Side Panel - Assembly Of
A458-5819	Valve Cover - Assembly Of
A458-5820	Fill Port - Perlite, Assembly & Detail
A458-5821 Sh 1 & 2	Electrical Schematic & Panel Layout

CCC 71-12 (ADDENDA)

ADDENDA TO
PETITION TO DEPARTMENT OF TRANSPORTATION
FOR SPECIAL PERMIT

200-POUND DIBORANE SHIPPING CONTAINER
DESIGN & PERFORMANCE

Developed Under Contract Number NASW 1827

ADDENDA: 14 JUNE 1971 - 18 SEPTEMBER 1972
ORIGINALLY SUBMITTED JUNE 1971

by

CALLERY CHEMICAL COMPANY
DIVISION OF MINE SAFETY APPLIANCES COMPANY
CALLERY, PENNSYLVANIA

16024

GALLERY CHEMICAL COMPANY
DIVISION OF MINE SAFETY APPLIANCES COMPANY

GALLERY, PENNSYLVANIA 16024 • PHONE (412) 538-3510

29 July 1971

Office of The Secretary of Transportation
Washington, D. C. 20590

Attention: Mr. James Grothe
Chief, Special Permits Branch
Office of Hazardous Materials

Dear Mr. Grothe:

This letter refers to the diborane shipping container developed and tested by Gallery Chemical Company under Contract Number NASH 1827. The data contained herein are an addition to the report submitted on 14 June 1971 in application for a special permit.

In telephone conversations with Mr. Paul Seay of your office, the limits of diborane net weight were discussed. We agreed that the basic permit should be issued for 200 ± 2 pounds net weight of diborane, but that an amendment should allow for smaller quantities of diborane.

Attached is a table showing the shipping time versus quantity in 20-pound increments of diborane net weight. As discussed in the initial presentation, the temperature of 0°C corresponds to a pressure approaching 400 psig or 80 percent of the maximum working pressure. Further, the temperature of -35°C was set as a somewhat arbitrary upper limit for the normal delivery time. On this basis the minimum quantity would be 100 pounds for a 15-day shipping permit. The container delivered in 15 days would arrive at a temperature approaching -35°C , and there would be an additional seven days before reaching 0°C .

Use of the container for quantities less than 200 pounds will be infrequent, but we feel this possibility should be covered by the regulations. We feel that 100 pounds is the minimum quantity for shipment under a 15 day permit. Further,

Mr. James Grothe

Page 2

we feel that use of the 15 day permit for a quantity range is preferable to a graduated shipping time, even if this limits the range somewhat. We invite your recommendations based on the attached data.

Very truly yours,

CALLERY CHEMICAL COMPANY
Division of MSA Company



K. W. Beahm
Project Leader

KWB/tp

Attachment

cc: Mr. R. M. Graziano, Agent
Bureau of Explosives
Association of American Railroads
1920 L. Street, N. W.
Washington, D. C. 20036

Mr. W. B. Powell, Technical Manager (2 copies)
Jet Propulsion Laboratory
California Institute of Technology
Building 125, Room 224
4800 Oak Grove Drive
Pasadena, California 91103

NASA
Headquarters Contracts Division
Washington, D. C. 20546
Attn: The Contracting Officer, Code DHC-6
Subject: Contract NASW-1827

Mr. Frank E. Compitello/Code RPL
NASA-OART
Liquid Propulsion Technology
600 Independence Avenue,
Washington, D. C. 20546

TABLE A-7

DIBORANE SHIPPING CONTAINER
EXTRAPOLATED TEST DATA

	Diborane. Weight, lbs	Time, Days	
		to -35°C	to 0°C
(1)	201.5	20.6	33.4
(2)	200	20.5	33.3
(2)	198	20.4	33.0
(2)	180	19.6	31.0
(2)	160	18.6	28.8
(2)	140	17.6	26.5
(2)	120	16.6	24.3
(2)	100	15.6	22.0
(2)	80	14.6	19.8

- (1) Actual test data
(2) Extrapolated test data

CALLERY CHEMICAL COMPANY
DIVISION OF MINE SAFETY APPLIANCES COMPANY

CALLERY, PENNSYLVANIA 16024 • PHONE (412) 538-3510

20 August 1971

Office of the Secretary of Transportation
Washington, D. C. 20590

Attention: Mr. James Grothe
Chief, Special Permits Branch
Office of Hazardous Materials

Dear Mr. Grothe:

This letter refers to the diborane shipping container developed and tested by Callery Chemical Company under Contract Number NASW 1827. The request contained herein is an addendum to the report dated 14 June 1971 and the followup letter dated 29 July 1971.

Our NASA contract cited above contains the requirement of a qualification shipping test, for which we plan to ship 200 pounds of methanol in the container. Mr. Paul Seay of your office has advised that a special permit is required to ship methanol in the diborane container, and we concur with his recommendation to include permission for qualification test shipments of methanol as a part of the diborane permit.

It is hereby requested, therefore, that you include in the diborane permit a statement to the effect:

"For container qualification test requirements, methanol (only) may be shipped in this container. Such test with methanol may be repeated as deemed necessary. Quantity range limitations cited for diborane will apply; however, requirement for pre-cooling is not applicable."

Since the initial qualification shipping test with methanol must be postponed until receipt of the special permit, we will appreciate anything you can do to expedite approvals of

Mr. James Grothe

- 2 -

the permit; thereby enabling Callery to complete the remaining work within the time limitations of our NASA contract.

Very truly yours,

CALLERY CHEMICAL COMPANY
DIVISION OF MSA COMPANY



K. W. Beahm
Project Leader

KWB:jp

cc: Mr. R. M. Graziano, Agent
Bureau of Explosives
Association of American Railroads
1920 L. Street, N. W.
Washington, D. C. 20036

Mr. W. B. Powell, Technical Manager (2 copies)
Jet Propulsion Laboratory
California Institute of Technology
4800 Oak Grove Drive
Pasadena, California 91103

NASA
Headquarters Contracts Division
Washington, D. C. 20546
Attn: The Contracting Officer, Code DHC-6
Subject: Contract NASW-1827

Mr. Frank E. Compitello/Code RPL
NASA-OART
Liquid Propulsion Technology
600 Independence Avenue
Washington, D. C. 20546

10 December 1971

Office of the Secretary of Transportation
Washington, D. C. 20590

Attention: Mr. James Grothe
Chief, Special Permits Branch
Office of Hazardous Materials

Dear Mr. Grothe:

Please refer to our letter of 27 October 1971 regarding the shipping classification of diborane, and to prior correspondence following our 14 June 1971 special permit application for the 200-pound container developed by Callery Chemical Company for NASA on Contract Number NASW-1827.

According to your telephone conversation with our Mr. A. J. Toering on 9 December 1971, we understand that DOT has ruled in favor of a dual classification for diborane: "Class A Poison" and "Flammable Compressed Gas". Please send written confirmation of this ruling in reply to our letter of 27 October 1971.

This will confirm our request that you proceed immediately to issue the special permit, which we understand has already been approved for motor vehicle shipment by the Federal Highway Administration. This will enable Callery to proceed with the methanol shipping test required by our NASA contract.

Further, Callery Chemical Company hereby requests that DOT consider approval of rail express as a mode of transportation for the 200-pound diborane shipping container. Although motor vehicle shipment is used whenever possible, there have been times when only rail shipment was possible to certain destinations. Rail freight is too slow for shipment under the ten-day or fifteen-day permits; therefore, rail shipment must be by rail express.

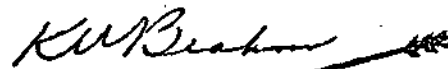
Mr. James Grothe

- 2 -

Please note that we do not want to holdup issue of the permit during consideration of the rail express question, which can be handled as a modification. Please expedite both these actions, because the NASA contract has already been seriously delayed.

Very truly yours,

CALLERY CHEMICAL COMPANY
DIVISION OF MSA COMPANY



K. W. Beahm

KWB:jp

cc: Mr. R. M. Graziano, Agent
Bureau of Explosives
Association of American Railroads
1920 L. Street, N. W.
Washington, D. C. 20036

Mr. W. B. Powell, Technical Manager (2 copies)
Jet Propulsion Laboratory
California Institute of Technology
Building 125, Room 224
4800 Oak Grove Drive
Pasadena, California 91103

NASA
Headquarters Contracts Division
Washington, D. C. 20546
Attn: The Contracting Officer, Code DHC-6
Subject: Contract NASW-1827

Mr. Frank E. Compitello/Code RPL
NASA-OART
Liquid Propulsion Technology
600 Independence Avenue
Washington, D. C. 20546

R. O. Voegtly, Safety Engineer

A. J. Toering, Manager - Marketing

H. W. Wilson, Manager - Engineering

3 March 1972

Office of the Secretary of Transportation
Washington, D. C. 20590

Attention: Mr. James Grothe
Chief, Special Permits Branch
Office of Hazardous Materials

Subject: DOT Special Permit No. 6522

Dear Mr. Grothe:

In accordance with our 3 March 1972 telephone conversation, attached is a table showing effect of diborane weight on shipping time for the container authorized under the subject permit. A part of these data had been presented in our letter of 29 July 1971, which became the basis for establishing 100 pounds as the minimum weight for a 15-day shipping permit.

The more complete data in the attached table show the reduction in time for quantities less than 100 pounds. As discussed in previous correspondence, the temperature of 0°C. corresponds to a pressure approaching 400 psig or 80 percent of the maximum working pressure. Further, the temperature of -35°C. was set as a somewhat arbitrary upper limit for the normal delivery time.

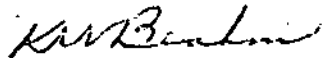
Based on the attached data we recommend a ten-day shipping time limit for quantities of 50 to 99 pounds and a five-day shipping time limit for quantities less than 50 pounds. The latter in effect is for quantities of 6 to 49 pounds, since 5 pounds above tare weight is allowed as "empty" for return (based on the fact that even total decomposition of 5 pounds of diborane would not result in overpressure of the container). All other terms of the permit, including precooling, should be made applicable.

Mr. Grothe

- 2 -

Please consider this a formal request for amendment of the subject permit to incorporate regulations for quantities less than 100 pounds. If there are any questions about the recommended graduated shipping times (5, 10, or 15 days) or about the supporting data, please feel free to call. In order to accomodate a planned NASA application, we would appreciate your action on this request for amendment prior to 15 April 1972.

Very truly yours,

CALLERY CHEMICAL COMPANY
DIVISION OF MSA COMPANY
K. W. Beahm
Project Leader

KWB:jp

Attachment (1)

cc: Mr. R. M. Graziano, Agent
Bureau of Explosives
Association of American Railroads
1920 L. Street, N. W.
Washington, D. C. 20036

Mr. W. B. Powell, Technical Manager (2 copies)
Jet Propulsion Laboratory
California Institute of Technology
Building 125, Room 224
4800 Oak Grove Drive
Pasadena, California 91103

TABLE A-8

DIBORANE SHIPPING CONTAINER
EXTRAPOLATED TEST DATA

	Diborane Weight, lbs.	Time, Days	
		to -35°C.	to 0°C.
(1)	201.5	20.6	33.4
(2)	200	20.5	33.3
(2)	198	20.4	33.0
(2)	180	19.6	31.0
(2)	160	18.6	28.8
(2)	140	17.6	26.5
(2)	120	16.6	24.3
(2)	100	15.6	22.0
(2)	80	14.6	19.8
(2)	60	13.7	17.5
(2)	50	13.2	16.4
(2)	40	12.7	15.2
(2)	20	11.7	13.0
(2)	5*	11.0	11.3

(1) Actual test data

(2) Extrapolated test data

* Would not exceed the maximum recommended operating pressure even on total decomposition.

CALLERY CHEMICAL COMPANY
DIVISION OF MINE SAFETY APPLIANCES COMPANY

CALLERY, PENNSYLVANIA 16024 • PHONE (412) 538-3510

15 March 1972

Office of the Secretary of Transportation
Washington, D. C. 20590

Attention: Mr. James Grothe
Chief, Special Permits Branch
Office of Hazardous Materials

Subject: DOT Special Permit No. 6522

Dear Mr. Grothe:

The subject permit has been reviewed in some detail by personnel at Callery Chemical Company and by representatives of NASA. In doing so, we have found that some wording in the permit leaves open the possibility of misinterpretation. This letter proposes, therefore, that certain sections be rewritten to prevent misuse of the permit. Specifically we invite your consideration of the following changes:

Paragraph 6

In line six, the sentence should stop after the word "insulation". The remainder of that sentence should be incorporated in a new sentence: "For diborane shipment, the dry ice chamber must contain not less than 108 pounds of dry ice on the date of shipment." (Or this sentence could be deleted since it is part of paragraph 7).

Insert in line ten (between the sentence ending with "Board." and the sentence beginning with "Except") the sentence previously given in sub-paragraph (7.b.), revised to: "Container must be equipped with an audio-visual alarm to indicate temperature above minus 35°C."

Paragraph 7

Reorganize this "SPECIAL PACKAGING REQUIREMENTS" paragraph to read as follows:

Mr. James Grothe

- 2 -

- a. All diborane shipments, including those which qualify as "empty" by subparagraph (c), must comply with the following:
- i. In addition to the flammable gas label and the poison gas label, each outside shipping container must bear a conspicuous label reading as follows:
"IF NOT DELIVERED BEFORE _____ CARRIER MUST ADVISE THE CALLERY CHEMICAL COMPANY, CALLERY, PENNSYLVANIA, ALSO THE BUREAU OF EXPLOSIVES, WASHINGTON, D.C., BY WIRE." The date inserted in the blank space on this label must not be in excess of the number of days prescribed herein from the date shipment is offered for transportation.
 - ii. Container must have not less than 108 pounds of dry ice in dry ice chamber on the day shipment is made.
 - iii. Container must reach destination within 15 days from date of shipment.
- b. All diborane shipments except those which qualify as "empty" by subparagraph (c) must also comply with the following:
- i. Filling is to be by weight only.
 - ii. Container must be precooled with liquid nitrogen to below minus 80°C. and excess nitrogen removed prior to charging with diborane.
- c. Container shipped as "empty" of diborane must also comply with the following:
- i. Must be verified to be "empty" by one of the following:
 - (a) The empty weight must not exceed the marked tare weight by more than 5 pounds, or
 - (b) Level of liquid diborane must be below the bottom of the dip tube. Loss of liquid seal will be evident by ability to vent gas pressure from the container (to user's tank or other proper vent system) through the dip tube.
 - ii. Pressure must be vented to between 25 and 50 psig at the time container is emptied.

Mr. James Grothe

- 3 -

d. Provisions for shipment of methanol are as follows:

- i. The only time methanol may be shipped is to satisfy contractor's qualification acceptance tests. Under these conditions neither the transit time restrictions nor the requirement for precooling apply.
- ii. Additionally, the requirements of subparagraphs (a), (b), and (c) of this paragraph and the entire paragraph (9) do not apply to methanol shipments.

Please understand that this request does not intend any change in the terms of the permit as approved; but rather the proposed wording should prevent misinterpretation of the terms agreed upon, thereby serving to benefit both DOT and those who use the permit.

If you desire to discuss this matter, please feel free to call or write.

Very truly yours,

CALLERY CHEMICAL COMPANY
DIVISION OF MSA COMPANY

K. W. Beahm
Project Leader

KWB:jp

cc: Mr. R. M. Graziano, Agent
Bureau of Explosives
Association of American Railroads
1920 L. Street, N. W.
Washington, D. C. 20036

Mr. W. B. Powell, Technical Manager (2 copies)
Jet Propulsion Laboratory
California Institute of Technology
Building 125, Room 224
4800 Oak Grove Drive
Pasadena, California 91103

cc: H. W. Wilson
A. J. Toering
R. O. Voegtly



CALLERY, PENNSYLVANIA 16024 • PHONE (412) 538-3510

28 March 1972

Office of the Secretary of Transportation
Washington, D. C. 20590

Attention: Mr. James Grothe
Chief, Special Permits Branch
Office of Hazardous Materials

Subject: DOT Special Permit No. 6522

Dear Mr. Grothe:

This letter is in regard to the various requests which have been made relative to the subject permit. In an effort to conclude our NASA contract at the earliest possible date, Callery hereby requests that action on these matters be expedited as follows:

On 3 March 1972 we requested authorization to ship diborane quantities less than 100 pounds under a graduated shipping time. A letter dated 20 March 1972 from Mr. R. M. Graziano, Director and Chief Inspector of Bureau of Explosives (AAR), reported that their Chief Chemist Charles Schultz felt additional test data were needed. Since we have some data which indirectly supports the information given, we feel the extrapolation could be defended technically; however, this would involve more time than is presently available. Further, it is possible that NASA may find it convenient to obtain measured data which bears directly on performance of the container with 50 to 60 pounds of diborane. In view of the above, therefore, please consider this a formal request to withdraw our request for authorization to ship quantities less than 100 pounds, with the understanding that Callery or NASA may reactivate the request by submitting additional data to support the recommendations.

Secondly there is our request of 15 March 1972 that certain sections of the permit be reworded to prevent any misinterpretation, without intending any change in the terms. Primarily this was aimed at clarification that 108 pounds of dry ice is

Mr. James Grothe

2

the requirement on the date of shipment, and not during all of the 15-day shipping time. We feel that this point must be clarified, whether or not you accept all of the recommended rewording.

Finally there remains your reply to our request of 10 December 1972 to consider approval of rail express as a mode of transportation. In a telephone conversation on 6 March 1972 Quentin Banks indicated that blanket approval may not be possible, but that single-trip exceptions might be considered if and when the situation arises. In any case we feel a written answer to our request is in order.

Your action in the above matters will permit Callery to close the NASA contract with no "loose ends". If there are any questions on these matters, please feel free to call or write.

Very truly yours,

CALLERY CHEMICAL COMPANY
DIVISION OF MSA COMPANY


K. W. Beahm

KWB:jp

cc: Mr. R. M. Graziano
Director and Chief Inspector
Bureau of Explosives, AAR
American Railroads Building
Washington, D. C. 20036

Mr. W. B. Powell, Technical Manager (2 copies)
Jet Propulsion Laboratory
California Institute of Technology
Building 125, Room 224
4800 Oak Grove Drive
Pasadena, California 91103



CALLERY, PENNSYLVANIA 16024 • PHONE (412) 538-3510

21 April 1972

Office of the Secretary of Transportation
Washington, D. C. 20590

Attention: Mr. James Grothe
Chief, Special Permits Branch
Office of Hazardous Materials

Subject: DOT Special Permit No. 6522

Dear Mr. Grothe:

The subject permit was discussed in your meeting with A. J. Toering on 13 April 1972, particularly in regard to the need for provision to ship a recoolled container.

Shipment of recoolled containers is discussed in the first attachment, with examples of cases where this may become necessary. The second attachment is an in-depth discussion of thermal performance for a recoolled container, including recommendations for safe shipping time; data are presented which were obtained after our original request for permit in June 1971. Finally, the third attachment is a proposed rewording of the permit to incorporate these and previous recommendations.

Please consider this a formal request to add to the terms of DOT Special Permit 6522 the following provision:

For shipment of a container recoolled to a
diborane pressure of 32 psig (corresponding
to diborane temperature of -70°C.) to reach
destination within

15 days for 175 to 200 lbs. B₂H₆
12 days for 100 to 174 lbs. B₂H₆

Further, we invite your consideration of the proposed wording of DOT Special Permit 6522 attached.

Other related matters still pending, summarized in Callery's letter of 28 March 1972, are as follows:

Mr. Grothe

- 2 -

(1) Rail express as a mode of transportation.

In a telephone conversation on 6 March 1972 Quentin Banks indicated that blanket approval may not be possible, but that single-trip exceptions might be considered if and when the situation arises. In any case we feel a written answer to our request is in order.

(2) Shipment of quantities less than 100 pounds.

This request was withdrawn by our letter of 28 March 1972, on the condition that it may be reactivated by submitting additional data to support the recommendations. NASA may obtain pertinent test data, and we presume you will consider it appropriate to reopen the question if additional data are provided.

(3) Rewording of special permit.

We feel that the dry ice specification must be clarified to 108 pounds on the date of shipment (not during all of the 15-day shipping time), whether or not you accept all of the recommended rewording.

We will appreciate your expeditious handling of these matters.

Very truly yours,

CALLERY CHEMICAL COMPANY
DIVISION OF MSA COMPANY



K. W. Beahm

KWB:jp

Attachments

cc: Mr. R. M. Graziano, Director & Chief Inspector
Bureau of Explosives (AAR)

Mr. W. B. Powell, Technical Manager
Jet Propulsion Laboratory

**DIBORANE SHIPPING CONTAINER
DOT SPECIAL PERMIT 6522**

SHIPMENT OF RECOOLED CONTAINERS

DOT Special Permit 6522 was issued with specific requirements for precooling, charging diborane, charging dry ice, and shipping; all in accordance with a prescribed schedule. Although this may cover most shipments, it is inevitable that the need will arise to ship a container after recooling with dry ice. Having lost the benefit of precooling, the safe shipping time is reduced somewhat. It becomes necessary, therefore, to define shipping limitations of a recool container to prevent misuse of the permit.

There have been cases where it became necessary to recool (add more dry ice) containers for shipment on our DOT Special Permit 970. The following will serve to illustrate:

1. A shipment of 40 pounds of diborane bound for the west coast via Rail Express was located in Chicago 7 days after leaving the Callery plant. Since it was apparent that the shipment would not reach its destination by the 10th day (time limit on Permit 970), the terminal manager in Chicago phoned Callery for instructions. A decision was made to return the shipment to the Callery plant where it was refilled with dry ice and reshipped via motor freight.
2. A shipment of 40 pounds of diborane to Sacramento, California arrived in Los Angeles on the 10th day. This shipment was located by Callery's shipping department in a routine follow-up check. Arrangements were made for Callery's Los Angeles representative to refill the container with dry ice at the truck terminal. Delivery was made to Sacramento without incident.
3. A shipment of 5 pounds of diborane for the Philadelphia, Penna. area was diverted to Newark, New Jersey and then returned to Philadelphia on the 11th day. When notified by the Philadelphia terminal manager, Callery sent a representative to refill the dry ice compartment. The shipment continued without incident.
4. A customer in the Philadelphia area received two cylinders of diborane the day after a portion of their plant was destroyed by fire. Since they were unable to use or dispose of the diborane, the containers were refilled with dry ice and returned to Callery via private truck.

DIBORANE SHIPPING CONTAINER
DOT SPECIAL PERMIT 6522

THERMAL PERFORMANCE - RECOOLING

The complete thermal storage test was concluded after 29 days by adding dry ice. Between 29 days and 62 days, dry ice was added periodically to observe recooling behavior. Between 62 days and 112 days, recooling was continued while diborane was removed as needed for in-plant uses. At 112 days the last 92.5 pounds of diborane was removed.

Callery's request for special permit, accompanied by design and performance specifications (CCC 71-12), was submitted in June 1971. This information included data through 6 June 1971, which corresponds to 45 days in the test. Subsequent data, not previously presented to DOT, were reported to NASA on Contract NASw 1827; and were therefore on record. These data are given here to support the terms of DOT Special Permit 6522.

Table 4 given here is a continuation of the Table 6 given on pages 27 and 28 of CCC 71-12. Columns for VI (voltage indicator) and LI (level indicator) were dropped as immaterial; while necessary data on True Temperature and Diborane Net Weight were added to Table 6. True Temperature is based on the indicated pressure, using vapor pressure data with correction for measured amount of non-condensables. Pressure vs temperature curves are given in Figure 4.

Recooling data are plotted in Figure 5 (six pages); curves are keyed to Table 4 by assignment of letters A through N. The most important result is that repeated dry ice addition established an equilibrium condition at about 32 psig, equivalent to -70°C . When dry ice weight approached 10 to 20 pounds, diborane temperature began to rise; but the -70°C . equilibrium was reestablished by adding more dry ice.

This equilibrium condition established by reicing is of some importance because of the potential need for shipment of a re-cooled container (as discussed in the previous section). It becomes necessary to define thermal performance of a re-cooled container, initially at -70°C ., compared to a pre-cooled container, initially at -80°C .

The data presented here are sufficient to make a reliable prediction of this thermal behavior, although none of the recooling tests was extended all the way to -35°C . Primary basis of the conclusions presented herein is recooling curve E, for several reasons. Recooling data for curves F through N involved diborane quantities less than 200 pounds, so are excluded at this point. Curves A and B

NOTE: In the original publication to DOT, Table 4 had incorrectly been referred to as "Table 6" at the beginning of paragraph 3 and in line 2 of paragraph 4. Correction made October 1972.

are excluded because they represent the cooldown period prior to establishing equilibrium required for shipment of a re-cooled container. Curve E extends all the way from 103 pounds of ice to the point where diborane temperature is rising, requiring much less of an extrapolation than curves C and D. Finally, the slopes of curves C and D are virtually identical, and may be shown to have the same overall heat transfer rate as measured during the complete storage test. On the other hand curve E represents a slightly higher heat transfer rate due to various conditions; and therefore using curve E is the most conservative or safest basis.

Page 2 of Figure 5 shows curve E rising to 49 psig with dry ice essentially gone; corresponding temperature is -62°C . Elapsed time from the start at -70°C . is 10.3 days. Thermal performance from -62°C . (and zero ice) to -35°C . and on up to 0°C . would be the same as measured in the earlier test, which differed only in its lower initial temperature. The earlier test data presented in CCC 71-12, are reproduced in part in Figure 6 on the same scale as Figure 5. In Table 2 on page 14 of CCC 71-12, it is recorded that -62°C . (49 psig) was reached at 14.3 days. It is apparent, therefore, that the difference in shipping time is four days (precooled container 14.3 days to -62°C . and zero ice, re-cooled container 10.3 days to -62°C . and zero ice). The combined data are shown in Figure 7.

Thus the time to any temperature above -62°C . would be four days less (for 200 pounds net weight of diborane), or:

	<u>Initial Temp., $^{\circ}\text{C}$.</u>	
	<u>-80</u>	<u>-70</u>
Days to -35°C .	20.5	16.5
Days to 0°C .	33.3	29.3

It must be emphasized that these calculations are for the worst case, or the safest assumptions. Other calculations made, but not included here, show the difference between precooled and re-cooled containers to be only one day, a more reasonable result; but we elect to take the most pessimistic result for greater margin of safety.

For diborane net weight between 100 and 200 pounds, the range authorized by DOT Special Permit 6522, the safest assumption would be to subtract four days from each previously reported value of storage/shipping time. Although this still gives a reasonable time for shipment, it may be shown that merely deducting four days would be unduly conservative. The following calculations will illustrate for 100 pounds.

Enthalpy Data (Figure 8)

10,220 BTU for 201.5# from -70 to -35°C.

8,000 BTU for 201.5# from -62 to -35°C.

2,200 BTU change during loss of dry ice

Measured rate between -62 and -35°C.

Time = 20.61 - 14.30 = 6.31 days

Rate = $\frac{8000 \text{ BTU}}{6.31 \text{ Days}} = 1268 \frac{\text{BTU}}{\text{day}}$

Correction for diborane net weight

 $(10,220) \frac{(100)}{(201.5)} = 5072 \text{ BTU}$

Use the safe assumption that the same 2200 BTU change will occur during loss of dry ice (10.3 days)

5072 - 2200 = 2852 BTU

Time after dry ice is gone (after 10.3 days)

 $\frac{2852 \text{ BTU}}{1268 \text{ BTU/day}} = 2.3 \text{ days}$

Total Time = 10.3 + 2.3 = 12.6 days

This time of 12.6 days to -35°C. (from initial temperature of -70°C.) compares to the 11.6 days one would obtain by the simple deduction of 4 days from the previously reported 15.6 days for an initial temperature of -80°C.

There would be no change in the time to go from -35°C. to 0°C., since these would be unaffected by initial temperature. Complete data would, therefore, be as follows:

B ₂ H ₆ Net Wt. lbs.	Time for Shipment or Storage, Days			
	Initially -70°C.		Initially -80°C.	
	to -35°C.	to 0°C.	to -35°C.	to 0°C.
200	16.5	29.3	20.5	33.3
175	15.6	26.7	19.3	30.4
150	14.6	24.1	18.1	27.6
125	13.6	21.6	16.8	24.8
100	12.6	19.0	15.6	22.0

This shows a 4-day difference at the full 200-pound loading, and a 3-day difference at the 100-pound loading.

As explained in CCC 71-12, 0°C. represents a maximum recommended operating condition approaching 400 psig in a vessel designed for 500 psig with pressure relief at 550 psig. The -35°C. was a somewhat arbitrary point selected for a container delivered within the time required by the shipping permit. For an initial temperature of -80°C., the permit authorizes 15-days for 100 pounds; a margin of 0.6 day to -35°C., and a margin of 7.0 days to 0°C. The same or greater margin would be allowed, for initial temperature of -70°C., if the permit were written for a shipping time graduated from 12 days with 100 pounds to 15 days with 200 pounds. For simplicity, however, we recommend only two shipping times, as follows:

-70°C. 15 days for 175 to 200 pounds
 12 days for 100 to 174 pounds

-80°C. 15 days for 100 to 200 pounds

It should be noted that the time margin to 0°C. (more important than the -35°C. point) will be at least equal, and in most cases greater than presently allowed.

In wording of the permit, confirmation of initial temperature by a pressure reading should be required. Not only is this more accurate, but it also prevents the possibility of shipping diborane containing excessive non-condensables, which could result in excessive pressures at acceptable temperatures.

Quantities less than 100 pounds were not included in the above discussion, since this is not presently authorized by the permit. It is worthy of note that the new data presented herein do relate to that question. In Figure 2 curves J, K, L, and M were obtained with diborane net weights of 96 to 107 pounds. Rate of ice loss is virtually identical to rates obtained with 200 pounds of diborane. These data are lacking a true temperature, however, because nitrogen introduced to pressure out the diborane negated the pressure-temperature conversion. They do confirm that dry ice lasts over ten days and lend some support to the extrapolations made for quantities less than 100 pounds. This point is made only by way of describing the data offered here, as this request does not cover anything below 100 pounds net weight of diborane.

TABLE A-9 PAGE 1 OF 6
TABLE 4 PAGE 1 OF 6

DIBORANE SHIPPING CONTAINER
POST-TEST RECOOL

TIME	DATE	DAYS	TI °C	PI PSIG	TRUE TEMP °C	VACUUM MICRONS	AMB. TEMP °F	SCALE READING LBS	DRY ICE, LBS.	B ₂ H ₆ WT. LBS.	REMARKS
0807	5/21/71	28.64	- 9.5	290	-12.3	17	72	2782.5	-0.5	201.5	
0815	5/21/71	28.65	- 8	290	-12.3	17.5	75	-	-	201.5	
1500	5/21/71	28.93	- 9	300	-11.0	20	82	2782.5	-0.5	201.5	End of Test
1520	5/21/71	28.94	-10	290	-12.3	19.5	80	2848.0	65.5	201.5	Dry Ice (A)
1000	5/22/71	29.72	-33	140	-36.0	10.5	70	2805.0	22.5	201.5	
1620	5/22/71	29.98	-39	125	-39.2	10.0	74	2796.0	13.5	201.5	
1630	5/22/71	29.99	-41	121	-40.2	9.5	74	2866.0	83.5	201.5	Dry Ice (B)
2245	5/23/71	31.25	-53	70	-54.5	6.0	68	2830.5	48.0	201.5	
0815	5/24/71	31.65	-59	60	-57.9	4.9	68	2822.5	40.0	201.5	
1600	5/24/71	31.97	-60	57	-59.0	5.9	80	2816.5	34.0	201.5	
0805	5/25/71	32.64	-62	50	-61.6	5.3	76	2805.0	22.5	201.5	
1550	5/25/71	32.96	-65	48	-62.5	5.5	78	2800.0	17.5	201.5	
0010	5/26/71	33.31	-63.5	43	-64.5	5.0	74	2796.0	13.5	201.5	
0807	5/26/71	33.64	-66	42	-65.0	4.1	69	2792.5	10.0	201.5	
1500	5/26/71	33.93	-65	40	-65.7	4.3	68	2790.0	7.5	201.5	
2200	5/26/71	34.22	-66	42	-65.0	4.3	71	2787.5	5.0	201.5	
0810	5/27/71	34.64	-66	42	-65.0	3.9	66	2785.0	2.5	201.5	
1535	5/27/71	34.95	-66	45	-63.7	4.5	71	2783.5	1.0	201.5	
0930	5/28/71	35.70	-55	52	-60.8	4.0	63	2782.5	0	201.5	
1500	5/28/71	35.93	-52	59	-58.2	5.2	72	2782.5	0	201.5	
1545	5/28/71	35.96	-60	55	-59.6	4.8	68	2867.5	85.0	201.5	Dry Ice (C)
2215	5/28/71	36.24	-63	47	-62.8	3.4	67	2860.5	78.0	201.5	
1030	5/29/71	36.74	-68	42	-65.0	2.6	65	2852.5	70.0	201.5	
1750	5/30/71	38.04	-71	37	-67.1	3.8	76	2837.0	54.5	201.5	
1140	5/31/71	38.79	-72	36	-67.5	4.2	74	2829.0	46.5	201.5	
2400	5/31/71	39.30	-72	34	-68.5	3.8	75	2824.0	41.5	201.5	
0810	6/1/71	39.64	-72	35	-68.0	3.6	72	2820.0	38.0	201.5	
1555	6/1/71	39.97	-72	35	-68.0	5.5	88	2817.0	34.5	201.5	
2050	6/1/71	40.17	-72	32	-69.7	5.4	83	2815.0	32.5	201.5	
0815	6/2/71	40.65	-72	32	-69.7	4.9	78	2810.0	27.5	201.5	
1600	6/2/71	40.97	-72	31	-70.0	4.9	77	2807.5	25.0	201.5	
0810	6/3/71	41.64	-72	31	-70.0	4.9	77	2801.5	19.0	201.5	

TABLE A9 PAGE 2 OF 6
TABLE 4 PAGE 2 OF 6

DIBORANE SHIPPING CONTAINER
POST-TEST RECOOL

TIME	DATE	DAYS	TI °C	PI PSIG	TRUE TEMP °C	VACUUM MICRONS	AMB. TEMP °F	SCALE READING LBS	DRY ICE, LBS.	B ₂ H ₆ WT LBS.	REMARKS
1600	6/3/71	41.97	-72	31	-70.0	6.0	88	2798.5	16	201.5	
0810	6/4/71	42.64	-72	31	-70.0	4.9	76	2792.5	10	201.5	
1405	6/4/71	42.89	-72	31	-70.0	7.0	90	2790.5	8	201.5	
1425	6/4/71	42.90	-72	31	-70.0	6.9	86	2853.5	71	201.5	Dry Ice (D)
1925	6/4/71	43.11	-73	33	-69.0	5.4	83	2850	67.5	201.5	
1145	6/5/71	43.79	-74	31	-70.0	4.4	80	2842	59.5	201.5	
1430	6/6/71	44.91	-74	31	-70.0	4.7	83	2833	50.5	201.5	
2345	6/6/71	45.29	-74	30	-70.6	3.2	70	2826.5	44	201.5	
0815	6/7/71	45.65	-74	30	-70.6	3.1	70	2823.5	41	201.5	
1530	6/7/71	45.95	-74	30	-70.6	5.5	82	2821.5	39	201.5	
2400	6/7/71	46.30	-76	29	-71.2	5.1	82	2817	34.5	201.5	
0810	6/8/71	46.64	-77	30	-70.6	4.8	80	2813.5	31	201.5	
1600	6/8/71	46.97	-77	30	-70.6	5.9	86	2810.5	28	201.5	
2100	6/8/71	47.18	-77	29	-71.2	4.8	79	2809	26.5	201.5	
0815	6/9/71	47.65	-77	30	-70.6	4.1	72	2804.5	22	201.5	
1600	6/9/71	47.97	-77	30	-70.6	5.5	82	2801.5	19	201.5	
2400	6/9/71	48.30	-77	31	-70.0	4.6	75	2799	16.5	201.5	
0810	6/10/71	48.64	-77	31	-70.0	4.0	75	2795.5	13	201.5	
1605	6/10/71	48.97	-55	35	-68.0	6.0	71	2790.5	8	201.5	TI Maintenance*
2345	6/10/71	49.29	-55	32	-69.7	5.1	77	2788	5.5	201.5	
0810	6/11/71	49.64	-55	32	-69.7	4.5	70	2786	3.5	201.5	
1000	6/11/71	49.72	-55	32	-69.7	5.0	76	2785.5	3	201.5	
1020	6/11/71	49.73	-55	32	-69.7	4.5	76	2885.5	103	201.5	Dry Ice (E)
1120	6/11/71	49.77	-58	32	-69.7	-	-	-	-	201.5	
1310	6/11/71	49.85	-56	32	-69.7	-	-	-	-	201.5	Meas. C.G.
1355	6/11/71	49.85	-55	33	-69.0	-	-	-	-	201.5	
1540	6/11/71	49.96	-58	31	-70.0	6.0	87	2884	101.5	201.5	
1945	6/11/71	50.12	-58	33	-69.0	6.2	86	2881	98.5	201.5	
1445	6/12/71	50.92	-58	31	-70.0	5.5	83	2870	87.5	201.5	
1705	6/13/71	52.01	-58	31	-70.0	6.1	90	2856	73.5	201.5	

*Note added October 1972

DIBORANE SHIPPING CONTAINER
POST-TEST RECOOL

TIME	DATE	DAYS	TI °C	PI PSIG	TRUE TEMP °C	VACUUM MICRONS	AMB. TEMP °F	SCALE READING LBS	DRY ICE, LBS.	B ₂ H ₆ WT LBS.	REMARKS
2400	6/13/71	52.30	-58	29	-71.2	4.9	80	2852	69.5	201.5	
0810	6/14/71	52.64	-58	29	-71.2	4.7	78	2848.5	66	201.5	
1550	6/14/71	52.96	-58	29	-71.2	5.2	86	2845	62.5	201.5	
2400	6/14/71	53.30	-57	28	-71.7	5.1	79	2841	58.5	201.5	
0810	6/15/71	53.64	-58	29	-71.2	4.7	78	2837	54.5	201.5	
1600	6/15/71	53.97	-58	29	-71.2	5.0	82	2833.5	51	201.5	
2400	6/15/71	54.30	-58	28	-71.7	4.8	77	2829.5	47	201.5	
0810	6/16/71	54.64	-58	29	-71.2	4.5	76	2826.5	44	201.5	
1555	6/16/71	54.97	-58	29	-71.2	5.3	84	2823.5	41	201.5	
2400	6/16/71	55.30	-58	30	-70.6	5.1	77	2819	36.5	201.5	
0810	6/17/71	55.64	-58	30	-70.6	4.0	71	2815.5	33	201.5	
1550	6/17/71	55.96	-58	30	-70.6	5.5	81	2812.5	30	201.5	
2400	6/17/71	56.30	-58	31	-70.0	5.2	79	2809	26.5	201.5	
0810	6/18/71	56.64	-58	31	-70.0	4.5	74	2806	23.5	201.5	
1605	6/18/71	56.97	-58	32	-69.7	7.0	92	2803	20.5	201.5	
2400	6/18/71	57.30	-58	32	-69.7	6.1	83	2799.5	17	201.5	
1650	6/19/71	58.00	-58	33	-69.0	7.7	95	2793	10.5	201.5	
1645	6/20/71	59.00	-53	37	-67.1	9.0	96	2785.5	3	201.5	
2345	6/20/71	59.29	-48	38	-66.6	9.1	88	2784	1.5	201.5	
0815	6/21/71	59.65	-38	42	-65.0	10	82	2783.5	1	201.5	
1520	6/21/71	59.94	-30	49	-62.1	11	86	2783.5	1	201.5	
1555	6/21/71	59.97	-30	49	-62.1	11	86	2848.5	66	201.5	Dry Ice (F)
2400	6/21/71	60.30	-48	42	-65.0	7.7	81	2840	57.5	201.5	
0810	6/22/71	60.64	-50	39	-66.2	6.5	76	2835	52.5	201.5	
1550	6/22/71	60.96	-50	39	-66.2	7.9	90	2830.5	48	201.5	
2345	6/22/71	61.29	-49	36	-67.5	6.8	82	2826	43.5	201.5	
0815	6/23/71	61.65	-50	35	-68.0	5.8	74	2821	38.5	201.5	
0900	6/23/71	61.68	-	-	-	-	-	2837.5	38.5	201.5	
0910	6/23/71	61.68	-50	35	-68.0	5.8	76	2837.5	38.5	201.5	Sampled
1300	6/23/71	61.85	-	42	-	-	-	2830	36	196.0	Rmv 5.5# B ₂

TABLE A-9 PAGE 4 OF 6
TABLE 4 PAGE 4 OF 6

DIBORANE SHIPPING CONTAINER
POST-TEST RECOOL

TIME	DATE	DAYS	TI °C	PI PSIG	TRUE TEMP °C	VACUUM MICRONS	AMB. TEMP °F	SCALE READING LBS	DRY ICE, LBS.	B ₂ H ₆ WT LBS.	REMARKS
1555	6/23/71	61.97	-51	42	*	7.4	92	2827.5	33.5	196.0	
2400	6/23/71	62.30	-52	41	-	6.3	82	2823	29	196.0	
0810	6/24/71	62.64	-52	41	-	5.5	78	2820	26	196.0	
1600	6/24/71	62.97	-52	41	-	7.9	95	2816	22	196.0	
2345	6/24/71	63.29	-52	40	-	6.7	86	2812	18	196.0	
0810	6/25/71	63.64	-52	41	-	6.9	80	2809	15	196.0	
1515	6/25/71	63.94	-52	41	-	6.9	91	2875	81	196.0	Dry Ice (G)
1745	6/26/71	65.04	-53	38	-	7.1	93	2857.5	63.5	196.0	
1745	6/27/71	66.04	-54	38	-	8.1	97	2845	-	196.0	
2400	6/27/71	66.30	-54	39	-	6.8	88	2842	48	196.0	
0810	6/28/71	66.64	-54	39	-	6.2	86	2838.5	44.5	196.0	
1555	6/28/71	66.97	-54	39	-	8.5	102	2829	40	192.0	Rmv 4.0# B ₂
0815	6/29/71	67.65	-54	39	-	6.5	88	2821	32	192.0	
1600	6/29/71	67.97	-54	39	-	8.6	103	2811	27	187.0	Rmv 5.0# B ₂
2345	6/29/71	68.29	-53	38	-	7.7	92	2808	24	187.0	
2345	6/30/71	69.29	-53	39	-	7.7	93	2797	13	187.0	
0810	7/1/71	69.64	-53	40	-	7.0	90	2793	9	187.0	
1600	7/1/71	69.97	-53	40	-	7.6	91	2790	6	187.0	
2345	7/1/71	70.29	-53	40	-	7.2	86	2787.5	3.5	187.0	
0815	7/2/71	70.65	-53	41	-	7.0	82	2785	1	187.0	
1515	7/2/71	70.94	-50	45	-	9.1	86	2874	90	187.0	Dry Ice (H)
2345	7/2/71	71.29	-51	42	-	5.7	76	2867	83	187.0	
1400	7/3/71	71.89	-51	40	-	5.8	80	2859	75	187.0	
1715	7/4/71	73.02	-52	39	-	6.2	84	2845	61	187.0	
1600	7/5/71	73.97	-52	39	-	6.3	85	2835	51	187.0	
2345	7/5/71	74.29	-52	39	-	5.7	80	2832	48	187.0	
0810	7/6/71	74.64	-52	39	-	5.2	76	2828.5	44.5	187.0	
1555	7/6/71	74.97	-52	54	-	7.1	86	2782.5	40.5	145.0	Rmv 42# B ₂
2400	7/6/71	75.30	-52	54	-	6.5	82	2777	35	145.0	
0810	7/7/71	75.64	-52	54	-	5.9	74	2774	32	145.0	
1525	7/7/71	75.94	-52	50	-	8.1	90	2732	28	107.0	Rmv 38# B ₂
2400	7/7/71	76.30	-52	52	-	6.8	84	2726	22	107.0	

*Unable to obtain temperature by pressure due to N₂ added to remove B₂H₆

DIBORANE SHIPPING CONTAINER
POST-TEST RECOOL

TIME	DATE	DAYS	TI °C	PI PSIG	TRUE TEMP °C	VACUUM MICRONS	AMB. TEMP °F	SCALE READING LBS	DRY ICE, LBS.	B ₂ H ₆ WT LBS.	REMARKS
0810	7/8/71	76.62	-52	50	-	6.0	78	2723	19	107.0	
1600	7/8/71	76.97	-52	50	-	8.8	96	2718	14	107.0	
2400	7/8/71	77.30	-52	52	-	7.5	88	2713	9	107.0	
0810	7/9/71	77.64	-52	52	-	8.0	88	2711	7	107.0	
1520	7/9/71	77.94	-52	52	-	8.5	94	2806	102	107.0	Dry Ice (J)
2400	7/9/71	78.30	-52	50	-	6.4	84	2799	95	107.0	
1630	7/10/71	78.99	-52	51	-	8.1	93	2789.5	85.5	107.0	
1645	7/11/71	80.00	-53	52	-	6.1	82	2778	74	107.0	
2400	7/11/71	80.30	-53	52	-	6.0	78	2774.5	70.5	107.0	
0810	7/12/71	80.64	-53	52	-	5.9	75	2770.5	66.5	107.0	
1600	7/12/71	80.97	-53	52	-	7.1	88	2766.5	62.5	107.0	
2400	7/12/71	81.30	-53	52	-	6.5	79	2762.5	58.5	107.0	
0810	7/13/71	81.64	-53	52	-	6.0	76	2759	55	107.0	
2345	7/13/71	82.29	-53	53	-	7.6	87	2751	47	107.0	
2200	7/14/71	83.22	-53	53	-	7.7	83	2741	37	107.0	
2345	7/15/71	84.29	-52	55	-	7.6	82	2730	26	107.0	
1515	7/16/71	84.94	-	-	-	-	-	2791	87	107.0	Dry Ice (K)
2215	7/16/71	85.23	-53	52	-	6.8	83	2786	82	107.0	
1445	7/17/71	85.92	-53	51	-	7.1	87	2777	73	107.0	
2230	7/18/71	87.24	-53	52	-	6.8	82	2762	58	107.0	
2145	7/19/71	88.21	-53	52	-	7.0	83	2751	47	107.0	
2200	7/20/71	89.22	-53	53	-	7.2	81	2740	36	107.0	
2215	7/21/71	90.23	-53	54	-	7.5	82	2729	25	107.0	
1605	7/22/71	90.97	-	-	-	-	-	-	-	99.5	Rmv 7.5# B ₂
2200	7/22/71	91.22	-53	53	-	7.7	85	2711	14.5	99.5	
2345	7/23/71	92.29	-53	51	-	5.8	78	2769	72.5	99.5	Dry Ice (L)
1800	7/24/71	93.05	-53	50	-	5.0	75	2759.5	63	99.5	
2045	7/25/71	94.17	-54	50	-	5.8	79	2746.5	50	99.5	
2345	7/26/71	95.29	-54	51	-	5.2	75	2733	36.5	99.5	
1650	7/27/71	96.00	-54	51	-	5.8	79	2727.5	31	99.5	
1645	7/28/71	97.00	-54	50	-	-	77	2718	21.5	99.5	
2330	7/29/71	98.00	-54	50	-	5.0	69	2706	9.5	99.5	

TABLE A-9 PAGE 6 OF 6

TABLE 4 PAGE 6 OF 6

DIBORANE SHIPPING CONTAINER
POST-TEST RECOOL

TIME	DATE	DAYS	TI °C	PI PSIG	TRUE TEMP °C	VACUUM MICRONS	AMB. TEMP °F	SCALE READING LBS	DRY ICE, LBS.	B ₂ H ₆ WT LBS.	REMARKS
1700	7/30/71	99.01	-57	49	-	4.9	74	2764	67.5	99.5	Dry Ice (M)
1610	7/31/71	99.98	-57	48	-	5.2	76	2753	56.5	99.5	
1700	8/1/71	101.01	-57	48	-	5.7	79	2742.5	46	99.5	
2350	8/2/71	102.30	-56	48	-	5.3	75	2730	33.5	99.5	
2345	8/3/71	103.29	-56	48	-	4.9	73	2720.5	24	99.5	
2400	8/4/71	104.30	-56	49	-	4.7	67	2711.5	15	99.5	Dry Ice (N)
2140	8/5/71	105.20	-56	49	-	5.6	72	2703	6.5	99.5	
1500	8/6/71	105.93	-	-	-	-	-	2697	0.5	99.5	
1510	8/6/71	105.93	-	-	-	-	-	2783	86.5	99.5	
2140	8/6/71	106.20	-56	49	-	5.4	73	2778.5	82	99.5	
1725	8/7/71	107.03	-57	48	-	6.2	81	2769	72.5	99.5	Rmv 2.0# B ₂
1700	8/8/71	108.01	-57	48	-	6.5	83	2759	62.5	99.5	
2200	8/9/71	109.22	-57	48	-	7.1	84	2746	49.5	99.5	
1600	8/10/71	109.97	-	-	-	-	-	-	-	97.5	
2200	8/10/71	110.22	-57	50	-	7.3	87	2731.5	37	97.5	
2100	8/11/71	111.18	-57	50	-	7.2	84	2712	17.5	97.5	Rmv 5.0# B ₂
1600	8/12/71	111.97	-	-	-	-	-	-	-	92.5	
2345	8/12/71	112.29	-57	49	-	6.6	78	2702	7.5	92.5	Unloaded
1305	8/13/71	112.85	-56	50	-	7.4	84	-	-	92.5	

FIGURE 4
TRUE TEMPERATURE BASED ON INDICATED PRESSURE

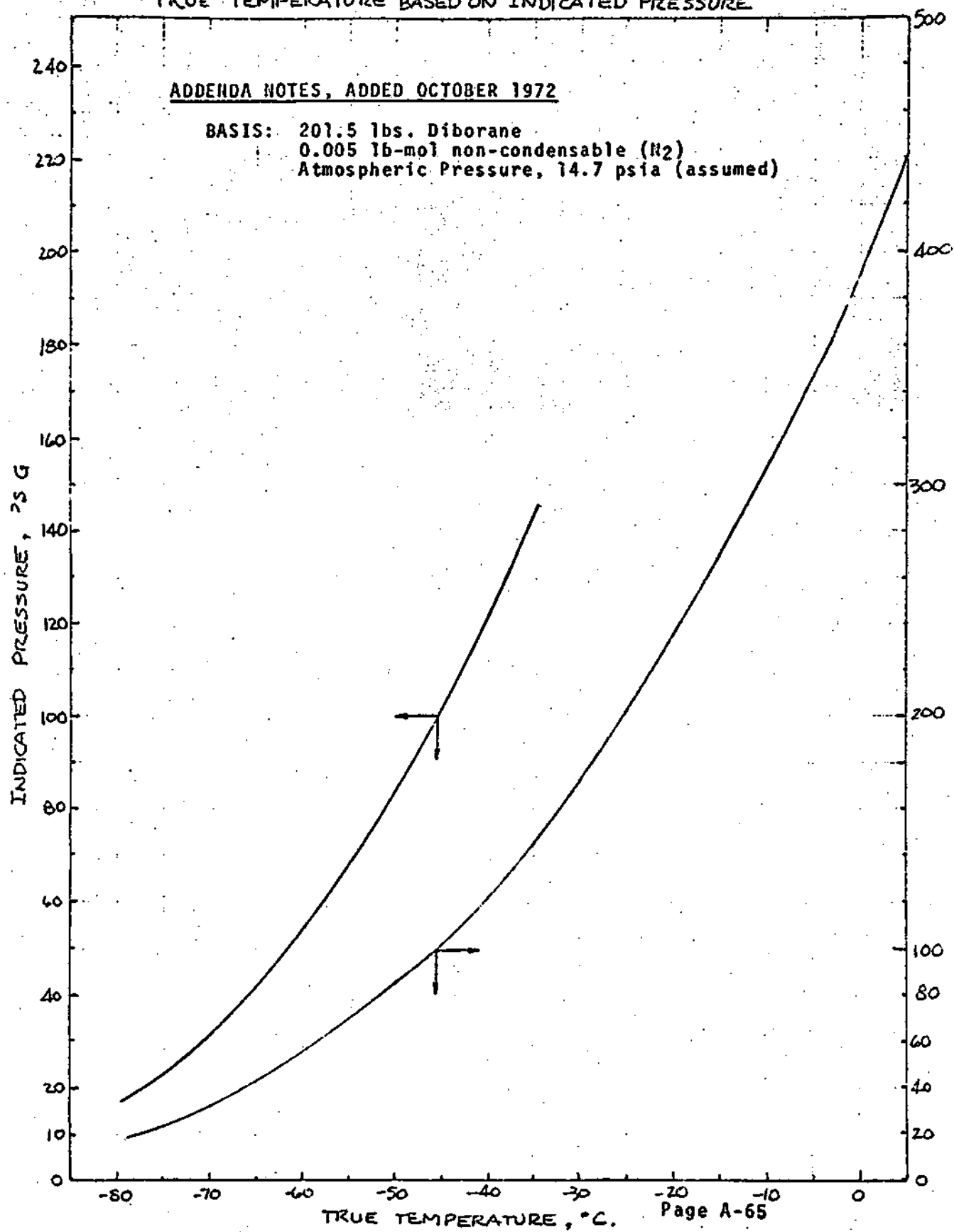


FIGURE 5
RECOOLING DATA

PAGE 1 OF 6 FIGURE A-5

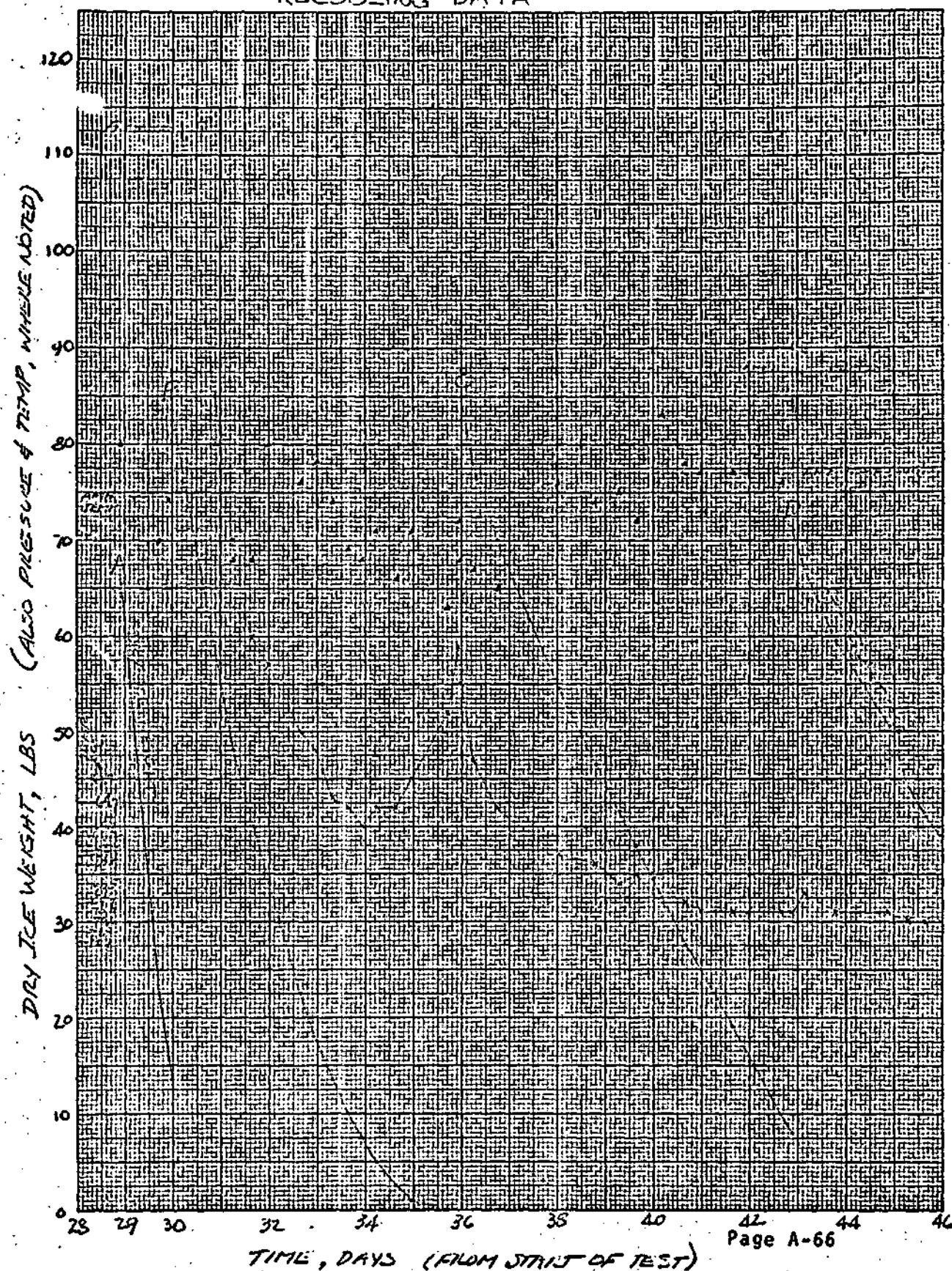


FIGURE 5
RECOOLING DATA

PAGE 2 OF 6 FIGURE A-5

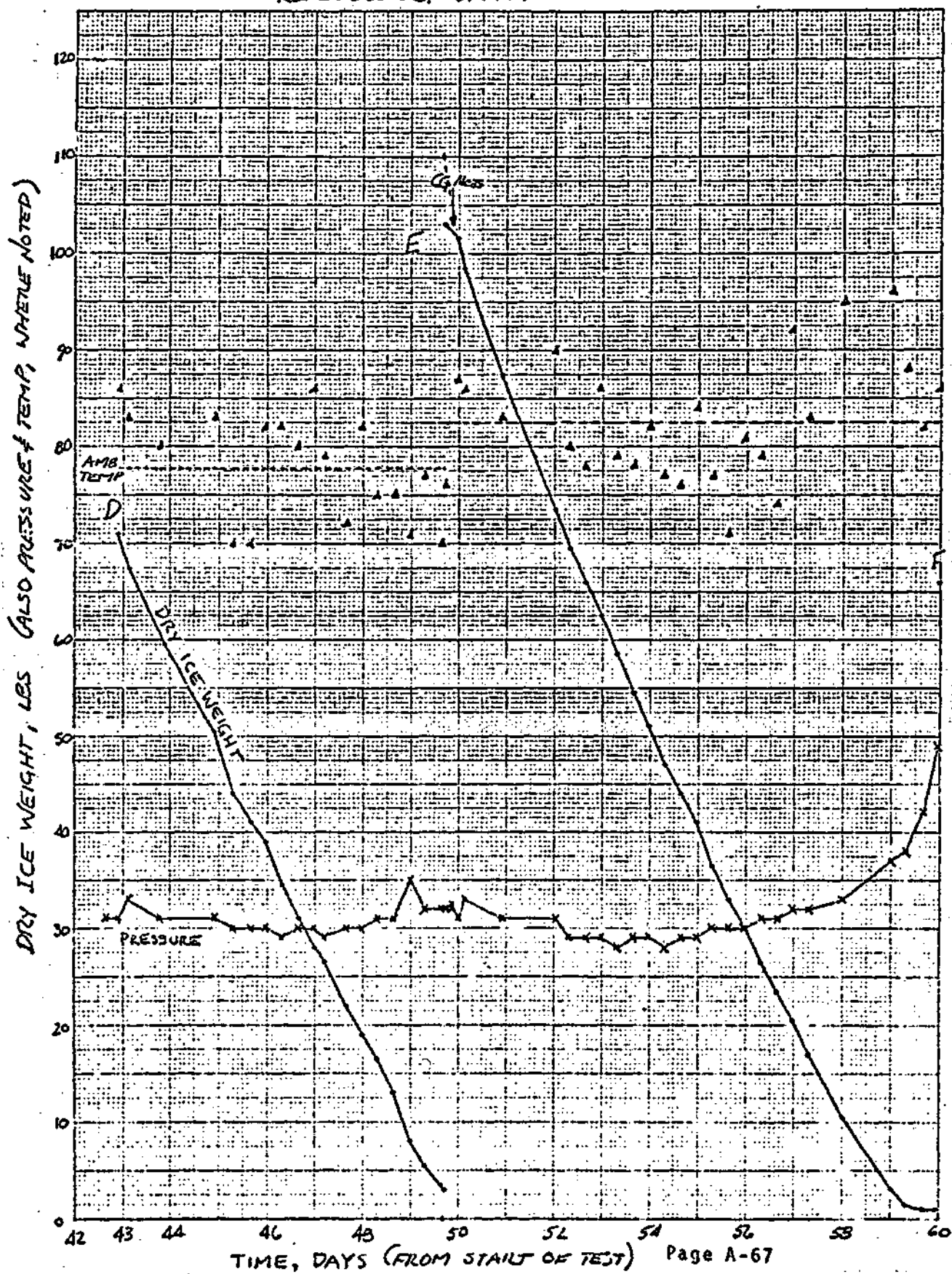


FIGURE 5 RECOOLING DATA PAGE 3 OF 6 FIGURE A-5

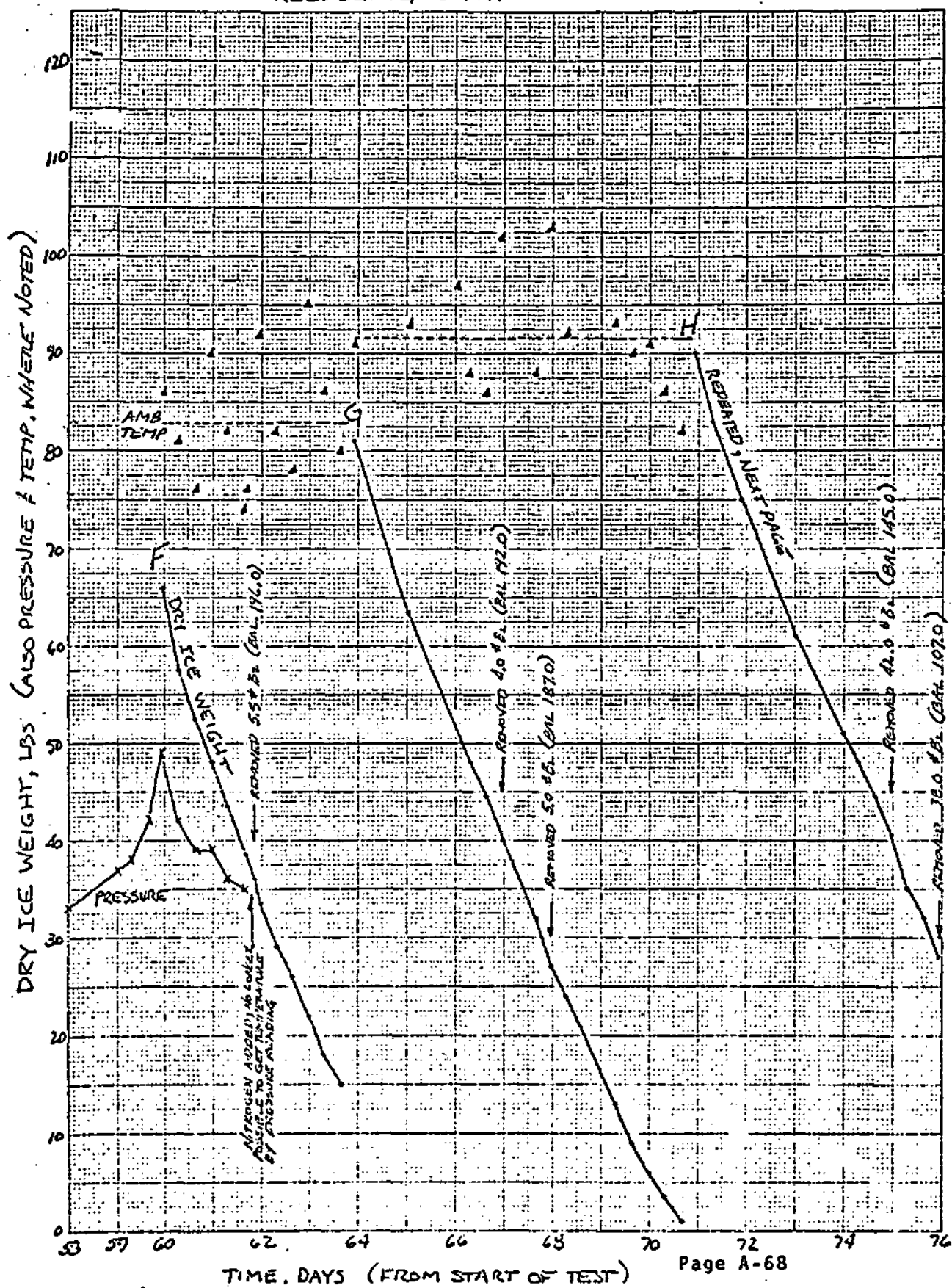


FIGURE 5
RECOOLING DATA

PAGE 4 OF 6

FIGURE A-5

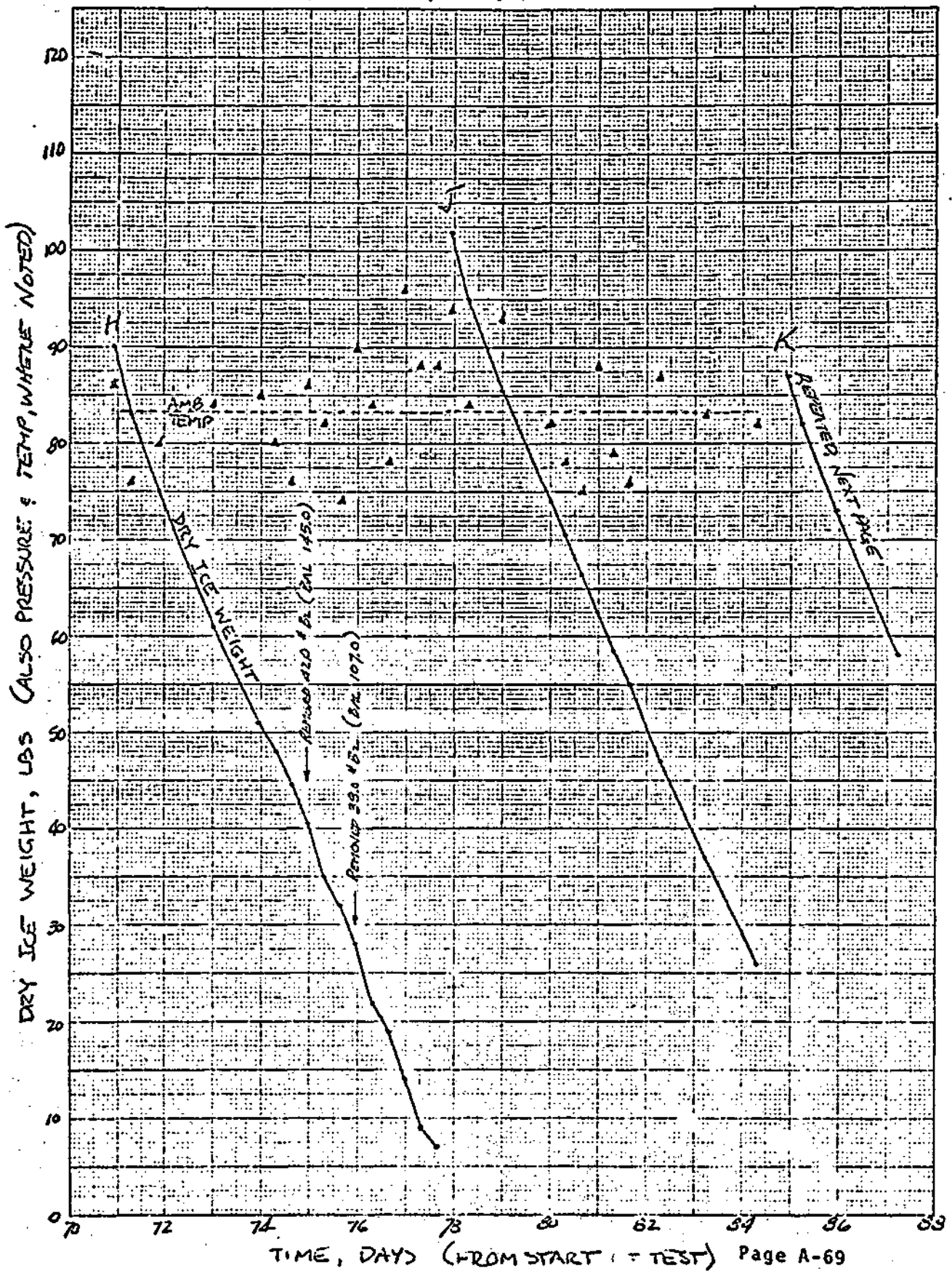


FIGURE 5
RECOILING DATA

PAGE 5 OF 6

FIGURE A-5

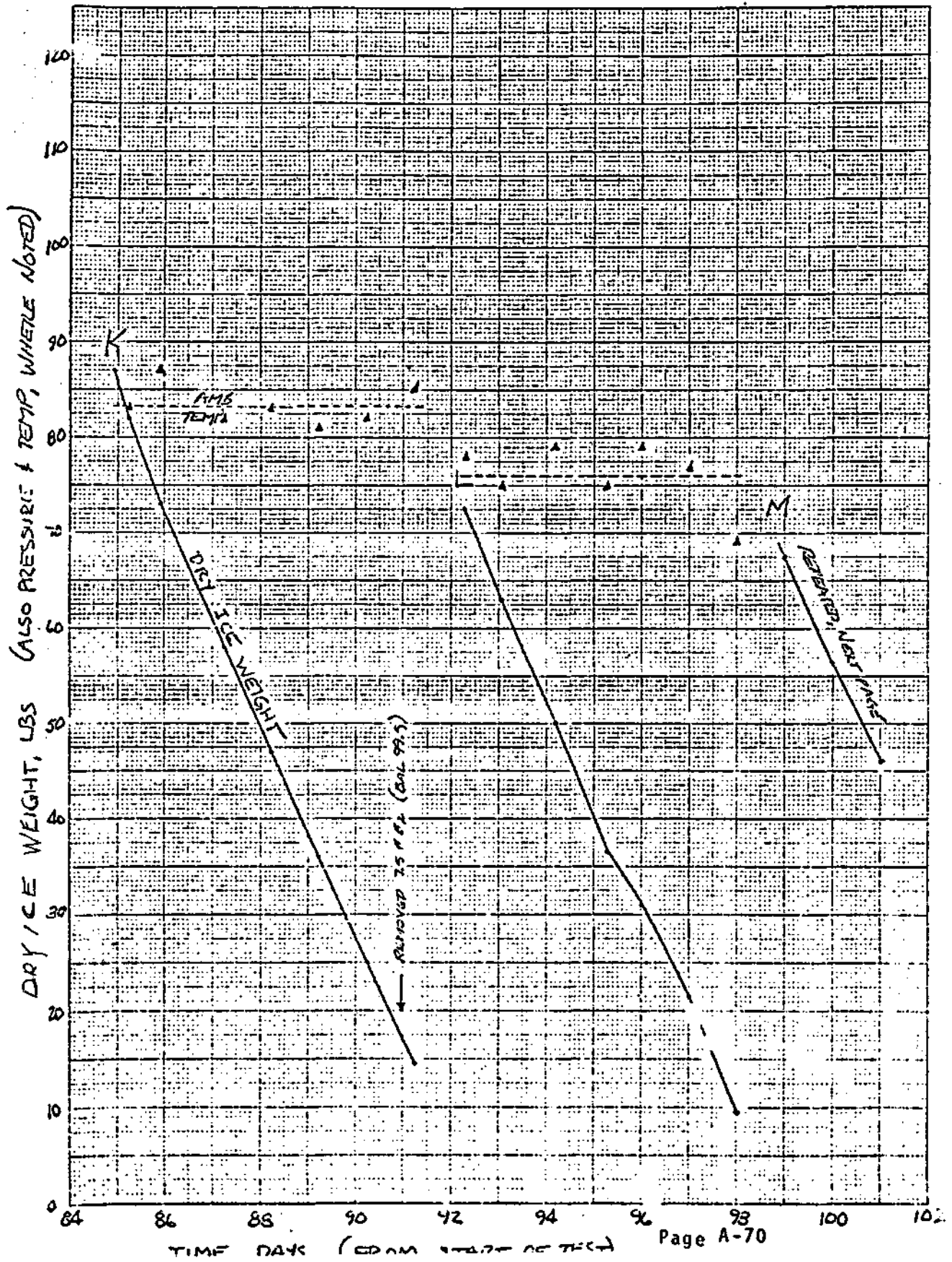


FIGURE 5
RECOOLING DATA

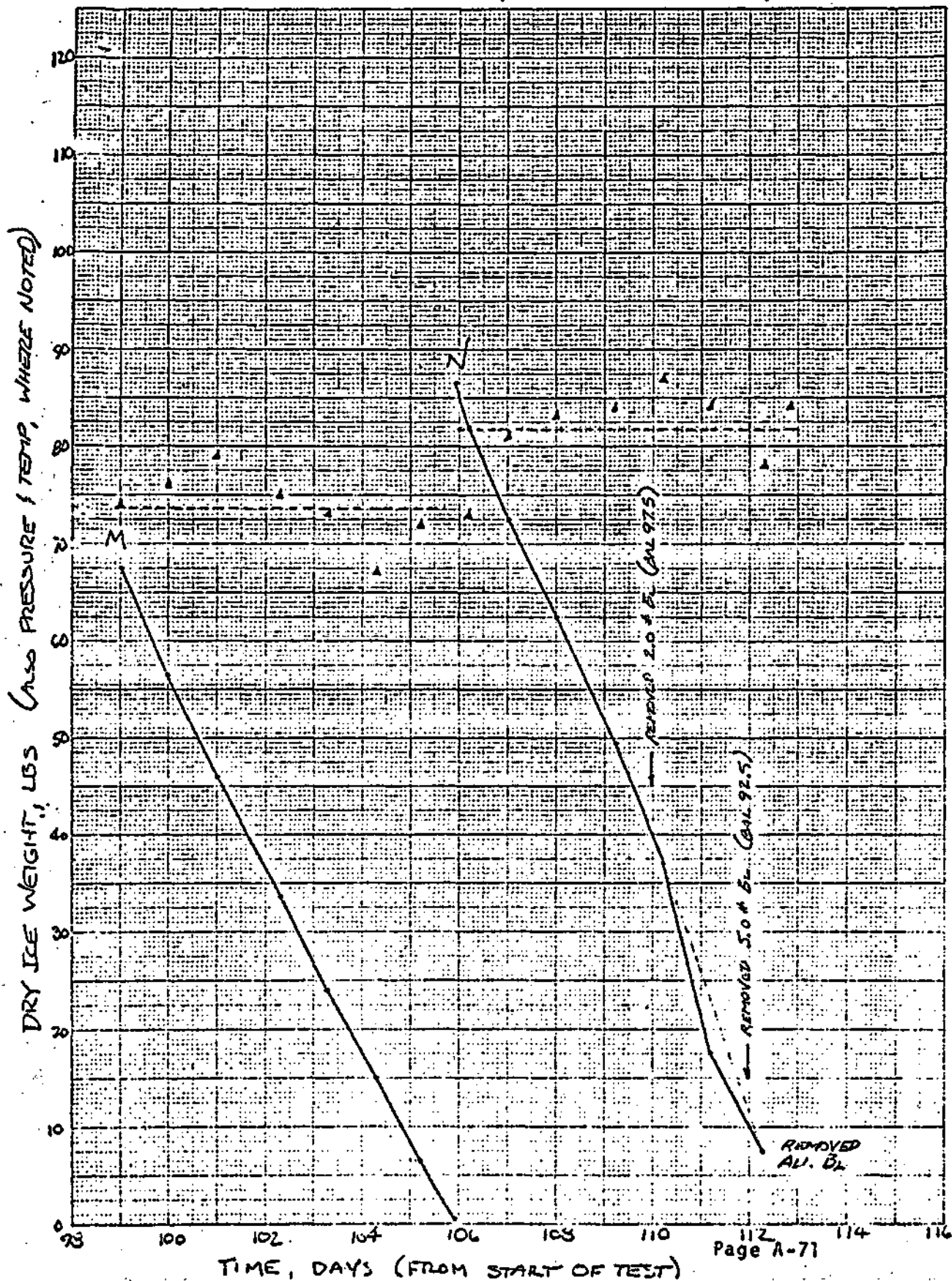


FIGURE 6
STORAGE TEST DATA

FIGURE A-6

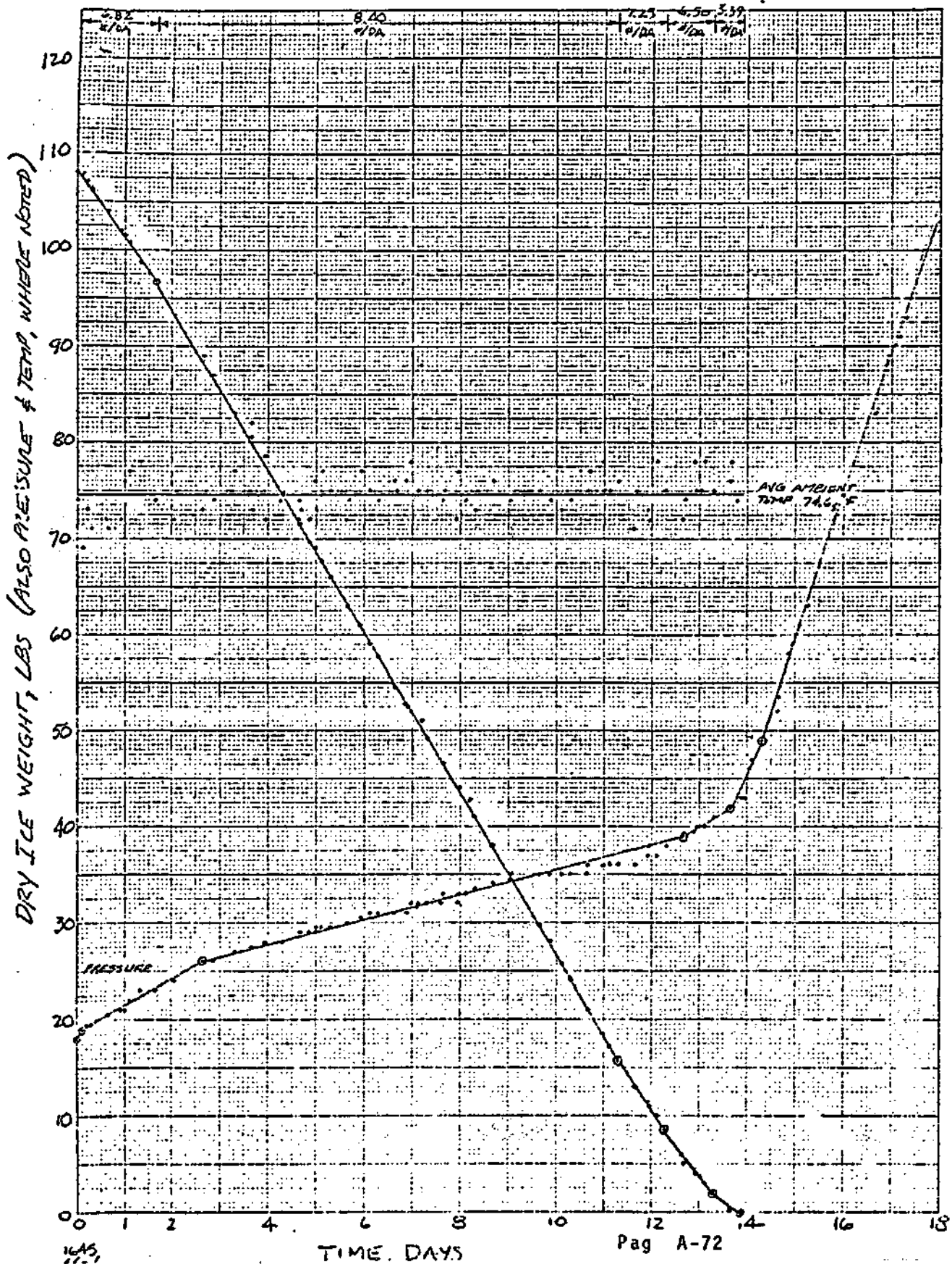


FIGURE 7 FIGURE A-7
PREDICTED THERMAL PERFORMANCE FOR
-70°C. INITIAL TEMPERATURE & 200 LBS B₂H₆

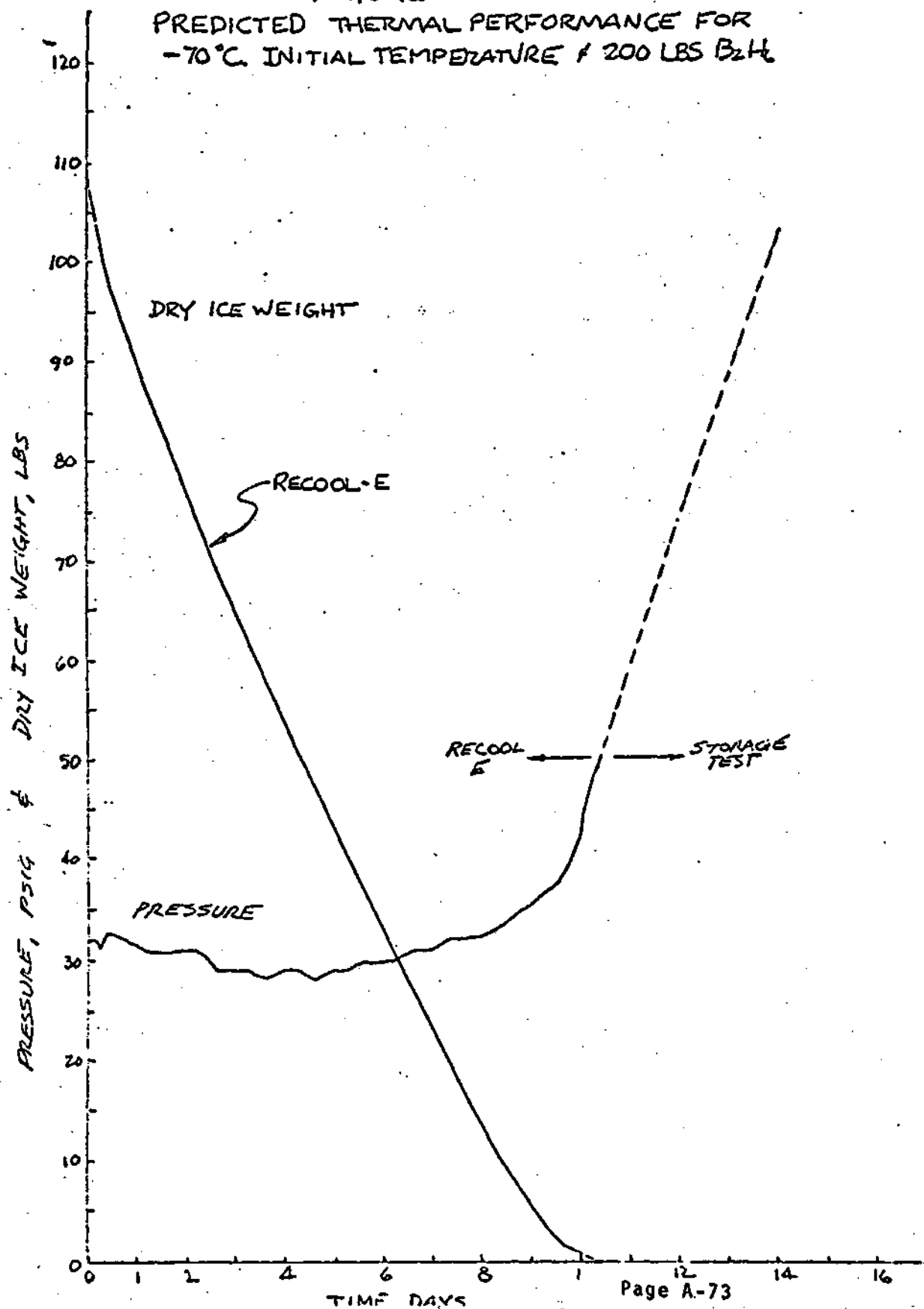
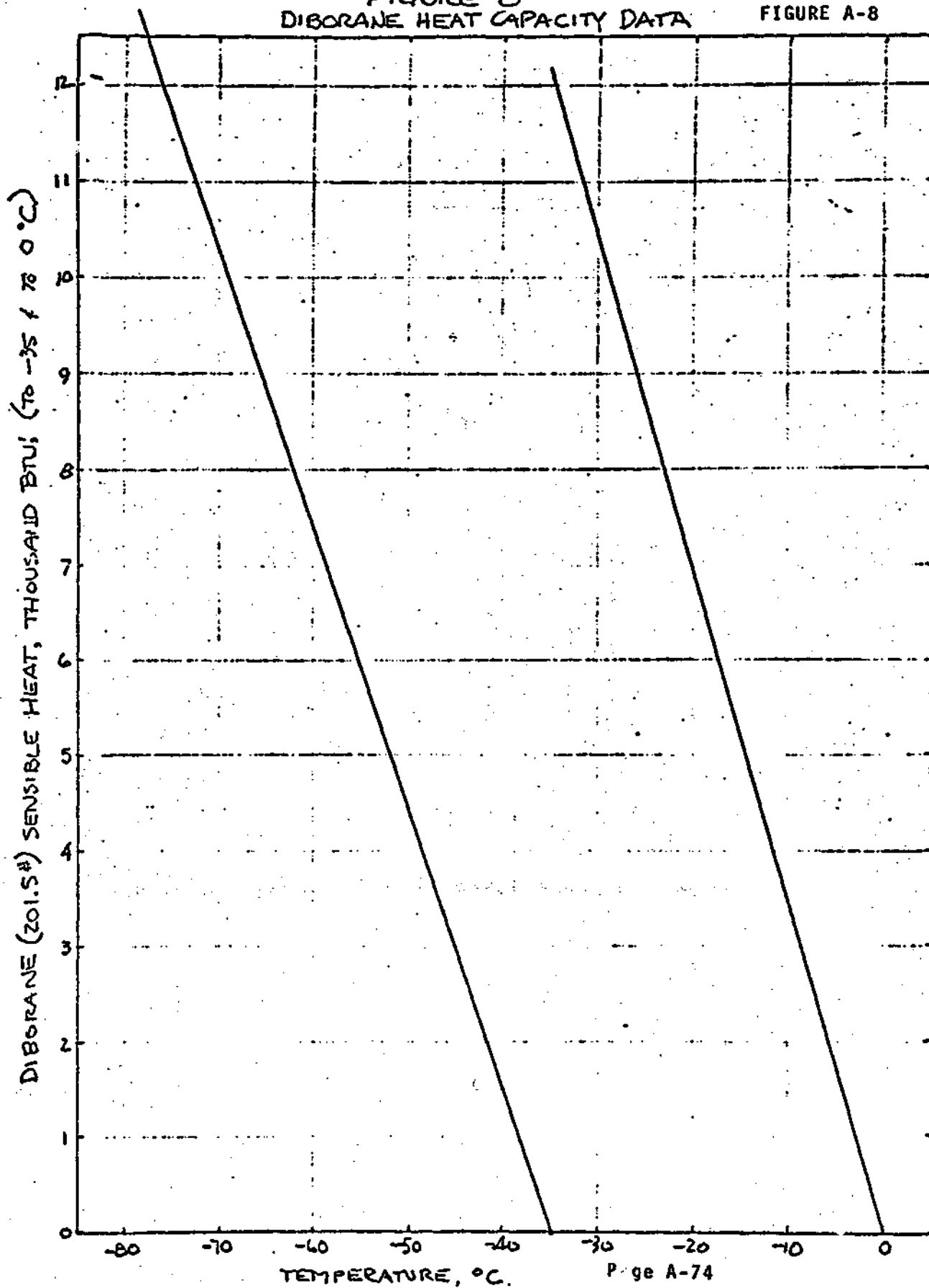


FIGURE 8
DIBORANE HEAT CAPACITY DATA

FIGURE A-8



SPECIAL PERMIT NO. 6522 [PROPOSED REWORDING]

This special permit is issued pursuant to 49 CFR 170.15 of the Department of Transportation (DOT) Hazardous Materials Regulations, as amended, to authorize shipments of a poisonous and compressed gas and a flammable liquid under conditions as prescribed herein. This permit does not relieve any shipper or carrier from compliance with any requirement of the DOT regulations, except as specifically provided for herein.

Standard special permit requirements and conditions relating to package markings, preparation of shipping papers, shipping experience reports, etc., are published in 49 CFR 171.6. These requirements are part of this special permit.

1. BASIS. June 14 and July 29, 1971 and April 21, 1972 petitions by Callery Chemical Company, Callery, Pa.

2. COMMODITY. Diborane or methanol (methyl alcohol).

3. PROPER SHIPPING NAME (49 CFR 172.5).

a. For Diborane: Both "Compressed gas, n.o.s. (Flammable)" and "Poisonous gas, n.o.s.".

b. For Methanol: "Methanol" or "Methyl alcohol".

4. REGULATION WAIVED. 49 CFR SS173.119, 173.304(a) (1) and 173.328.

5. AUTHORIZED SHIPPER. The petitioner identified above and its customers that are registered with this Board.

6. PACKAGING PRESCRIBED. In a specially designed 36-inch diameter spherical inner tank having a maximum working pressure of 500 psig containing not less than 100 pounds nor more than 200 pounds of product. Inner tank is surrounded (except for the dry ice chamber) by a 48-inch diameter cylindrical shell containing perlite insulation. For diborane shipment, the dry ice chamber must contain not less than 108 pounds of dry ice on the date of shipment. Container must be fabricated, and assembled in accordance with details included in CVI Corporation's drawings A458-5800 through A458-5821 on file with this Board. Container must be equipped with an audio-visual alarm to indicate temperature above -3°C. Except as otherwise provided herein, the container must be qualified and prepared for shipment in accordance with the petitioner's design and performance specifications on file with this Board.

7. SPECIAL PACKAGING REQUIREMENTS.

a. ALL diborane shipments, including those which qualify as "empty" by subparagraph (c), must comply with the following:

i. In addition to the flammable gas label and the poison gas label, each outside shipping container must bear a conspicuous label reading as follows; "IF NOT DELIVERED BEFORE _____ CARRIER MUST ADVISE THE CALLERY CHEMICAL COMPANY, CALLERY, PENNSYLVANIA, ALSO THE BUREAU OF EXPLOSIVES, WASHINGTON, D.C., BY WIRE." The date inserted in the blank space on this label must not be in excess of the number of days prescribed herein from the date shipment is offered for transportation.

ii. Container must have not less than 108 pounds of dry ice in dry ice chamber on the day shipment is made.

b. All diborane shipments except those which qualify as "empty" by subparagraph (c) must ALSO comply with the following:

i. Filling is to be by weight only.

ii. Containers precooled to minus 80°C. must reach destination within 15 days from date of shipment. True temperature at or below -80°C. must be confirmed by pressure of 18 psig or lower.

iii. Containers precooled or recooled to minus 70°C. must reach destination within the following schedule.

(a) 15 days from date of shipment when diborane net weight is 175 to 200 pounds.

(b) 12 days from date of shipment when diborane net weight is 100 to 174 pounds.

True temperature at or below -70°C. must be confirmed by pressure of 32 psig or lower.

c. Container shipped as "empty" of diborane must ALSO comply with the following:

i. Must be verified to be "empty" by one of the following:

(a) The empty weight must not exceed the marked tare weight by more than 5 pounds, or

(b) Level of liquid diborane must be below the bottom of the dip tube. Loss of liquid seal will be evident by ability to vent gas pressure from the container (to user's tank or other proper vent system) through the dip tube.

ii. Pressure must be vented to between 25 and 50 psig at the time container is emptied.

d. Provisions for shipment of methanol are as follows:

i. The only time methanol may be shipped is to satisfy contractor's qualification acceptance tests. Under these conditions neither the transit time restrictions nor the requirement for cooling apply.

ii. Additionally, the requirements of subparagraphs (a), (b), and (c) of this paragraph and the entire paragraph (9) do not apply to methanol shipments.

8. MODES OF TRANSPORTATION AUTHORIZED. Motor vehicle.

9. SPECIAL TRANSPORTATION REQUIREMENTS.

a. A copy of this permit, kept current, must be carried aboard each motor vehicle.

b. Shipments of diborane made under the terms of this permit must be delivered within the time prescribed in paragraph 7(b).

c. Each shipping paper must show thereon, following the commodity description, the notation, appropriately executed:

DOT SPECIAL PERMIT NO. 6522
DATE OF SHIPMENT _____
IF NOT DELIVERED BEFORE _____
DAYS CARRIER MUST ADVISE BUREAU
OF EXPLOSIVES, WASHINGTON, D.C.
BY WIRE.

d. Each shipper must require acknowledgement of receipt of shipment from consignee by wire, to be confirmed in writing, and must promptly notify the Bureau of Explosives (AAR) of any such shipment not received at destination within two days after shipment is due.

e. Any common carrier by motor vehicle transporting diborane under the terms of this permit must be specifically approved by the Federal Highway Administration.

10. REPORTING REQUIREMENTS. Any incident involving loss of contents of the package must be reported to this Board as soon as practicable.

11. EXPIRATION DATE. September 15, 1972.

Issued at Washington, D. C. :

W. R. Fiste
For the Administrator
Federal Highway Administration

(DATE)

Address all inquiries to: Secretary, Hazardous Materials Regulations Board, U.S. Department of Transportation, Washington, D. C. 20590.
Attention: Special Permits.

Dist: a, d,

A. J. Toering
R. O. Voegtly

CALLERY CHEMICAL COMPANY
DIVISION OF MINE SAFETY APPLIANCES COMPANY

CALLERY, PENNSYLVANIA 16024 • PHONE (412) 538-3510

24 April 1972

Office of the Secretary of Transportation
Washington, D. C. 20590

Attention: Mr. James Grothe
Chief, Special Permits Branch
Office of Hazardous Materials

Subject: DOT Special Permit No. 6522

Dear Mr. Grothe:

You have no doubt figured out that our letters crossed in the mails, which obsoletes a part of our 21 April 1972 letter.

First Revision to Special Permit No. 6522 (just received) covers all items except provision for recooling, which was the main thrust of our most recent letter. Attachments one and two to that letter are still applicable, as a followup to your meeting with A. J. Toering on 13 April 1972.

To prevent confusion, you will find attached to this letter a list of modifications necessary to incorporate recooling into First Revision of DOT Special Permit 6522. This will replace attachment three to our 21 April 1972 letter.

We appreciate your cooperation in this joint effort to achieve safe and practical shipment of diborane, and we look forward to your reply on the recooling provisions.

Very truly yours,

CALLERY CHEMICAL COMPANY
DIVISION OF MSA COMPANY


K. W. Beahm

KWB:jp

Attachments

cc: Mr. R. M. Graziano, Director & Chief Inspector
Bureau of Explosives (AAR)

Mr. W. B. Powell, Technical Manager
Jet Propulsion Laboratory

Modifications Necessary to Incorporate
Recooling Provisions in

SPECIAL PERMIT NO. 6522
FIRST REVISION

After "1. BASIS." delete present wording and replace with
"April 21, 1972 petition by Callery Chemical Company, Callery,
Pa."

Delete subparagraph 7(a)iii and subparagraph 7(b)ii. In their
place, add subparagraphs 7(b)ii and 7(b)iii; as follows:

7(b)ii Containers precooled to minus 80°C. must reach
destination within 15 days from date of shipment. True
temperature at or below -80°C. must be confirmed by
pressure of 18 psig or lower.

7(b)iii Containers precooled or recoolled to minus 70°C.
must reach destination within the following schedule.

(a) 15 days from date of shipment when diborane net
weight is 175 to 200 pounds.

(b) 12 days from date of shipment when diborane net
weight is 100 to 174 pounds.

True temperature at or below -70°C. must be confirmed
by pressure of 32 psig or lower.

In subparagraph 9(b) delete "within 15 days from date of
shipment", and in its place add "within the time prescribed
in subparagraph 7(b)".

bc: H. W. Wilson
A. J. Toering
R. O. Voegtly

CALLERY CHEMICAL COMPANY
DIVISION OF MINE SAFETY APPLIANCES COMPANY

CALLERY, PENNSYLVANIA 16024 • PHONE (412) 538-3510

30 May 1972

Office of the Secretary of Transportation
Washington, D. C. 20590

Attention: Mr. James Grothe
Chief, Special Permits Branch
Office of Hazardous Materials

Subject: DOT Special Permit No. 6522

Dear Mr. Grothe:

During review of First Revision to the subject permit, one point has arisen which should be clarified.

In subparagraph 7(c)i, terms are given to define an "empty" container. The statement "Pressure should be vented to between 25 and 50 psig at the time container is emptied." is incorporated in (b) of the subparagraph cited; whereas, this venting is a requirement for safety in either case, (a) or (b). It is proposed, therefore, that the statement about venting be moved to subparagraph 7(c)ii, making it a requirement of an "empty" container whether defined by (a) or (b) of subparagraph 7(c)i.

Perhaps the above change could be made when you incorporate provisions for shipment of a recooled container, as requested in our letters of 21 April 1972 and 24 April 1972.

Very truly yours,

CALLERY CHEMICAL COMPANY
DIVISION OF MSA COMPANY

KW Beahm

K. W. Beahm

KWB:jp

cc: Mr. R. M. Graziano, Director & Chief Inspector
Bureau of Explosives (AAR)

Mr. W. B. Powell, Technical Manager
Jet Propulsion Laboratory

cc: C. E. Iman
H. W. Wilson
A. J. Toering
R. O. Voegtly

CALLERY CHEMICAL COMPANY
DIVISION OF MINE SAFETY APPLIANCES COMPANY

CALLERY, PENNSYLVANIA 15024 • PHONE (412) 533-3510

30 August 1972

Office of the Secretary of Transportation
Washington, D. C. 20590

Attention: Mr. James Grothe
Chief, Special Permits Branch
Office of Hazardous Materials

Subject: DOT Special Permit No. 6522

Dear Mr. Grothe:

In accordance with our telephone conversation today, attached are the proposed revisions to the subject permit. This incorporates the provision for 15-day limit on shipment of an "empty" container and defines the recommended method of cooling. The word "recooling" has been deleted in favor of "cooling"; because it is anticipated that the minus 70°C. may be used as the initial temperature on some shipments, as well as possible use for recooling containers delayed in transit.

In addition, since the expiration date of the subject permit presently is September 15, 1972, it is hereby requested that this permit be renewed to extend the expiration date by one year, to September 15, 1973.

Very truly yours,

CALLERY CHEMICAL COMPANY
DIVISION OF MSA COMPANY



K. W. Beahm

KWB:jp

Attachment

cc: Mr. R. M. Graziano, Director & Chief Inspector
Bureau of Explosives (AAR)

Mr. W. B. Powell, Technical Manager
Jet Propulsion Laboratory

Recommended Changes
from FIRST REVISION
to SECOND REVISION
of SPECIAL PERMIT No. 6522

After "1. BASIS." delete present wording and replace with "April 21, 1972 and August 30, 1972 petitions by Callery Chemical Company, Callery, Pa."

Delete subparagraph 7(a)iii and subparagraph 7(b)ii. In their place, add subparagraphs 7(b)ii and 7(b)iii; as follows:

7(b)ii Containers precooled to minus 80°C. with liquid nitrogen must reach destination within 15 days from date of shipment. True temperature at or below -80°C. must be confirmed by pressure of 18 psig or lower on date of shipment.

7(b)iii Containers cooled to minus 70°C. with dry ice (or liquid nitrogen) must reach destination within the following schedule:

(a) 15 days from date of shipment when diborane net weight is 175 to 200 pounds.

(b) 12 days from date of shipment when diborane net weight is 100 to 174 pounds.

True temperature at or below -70°C. must be confirmed by pressure of 32 psig or lower on date of shipment.

Move the statement "Pressure should be vented to between 25 and 50 psig at the time container is emptied." from subparagraph 7(c)i(b) to subparagraph 7(c)ii, which has the effect of making it applicable to both 7(c)i(a) and 7(c)i(b).

Add subparagraph 7(c)iii, stating "Containers shipped as empty must reach destination within 15 days from date of shipment."

In subparagraph 9(b) delete "within 15 days from date of shipment", and in its place add "within the time prescribed in subparagraph 7(b)".

DEPARTMENT OF TRANSPORTATION
HAZARDOUS MATERIALS REGULATIONS BOARD
WASHINGTON, D.C. 20590

SPECIAL PERMIT NO. 6522
SECOND REVISION
(COMPLETE)

This special permit is issued pursuant to 49 CFR 170.15 of the Department of Transportation (DOT) Hazardous Materials Regulations, as amended, to authorize shipments of a poisonous and compressed gas; and a flammable liquid under conditions as prescribed herein. This permit does not relieve any shipper or carrier from compliance with any requirement of the DOT regulations, except as specifically provided for herein.

Standard special permit requirements and conditions relating to package markings, preparation of shipping papers, shipping experience reports, etc., are published in 49 CFR 171.6. These requirements are part of this special permit.

1. BASIS. April 21, 1972 and August 30, 1972 petitions by Callery Chemical Company, Callery, Pa.

2. COMMODITY. Diborane or methanol (methyl alcohol).

3. PROPER SHIPPING NAME (49 CFR 172.5).

a. For Diborane: Both "Compressed gas, n.o.s. (Flammable)" and "Poisonous gas, n.o.s.".

b. For Methanol: "Methanol" or "Methyl alcohol".

4. REGULATION WAIVED. 49 CFR SS173.119, 173.304(a) (1) and 173.328.

5. AUTHORIZED SHIPPER. The petitioners identified above and their customers who register their identity with and receive acknowledgement from this Board and have a copy of the special permit.

6. PACKAGING PRESCRIBED. In a specially designed 36-inch diameter spherical inner tank having a maximum working pressure of 500 psig containing not less than 100 pounds nor more than 200 pounds of product. Inner tank is surrounded (except for the dry ice chamber) by a 48-inch diameter cylindrical shell containing perlite insulation. Container must be equipped with an audio-visual alarm to indicate temperature above minus 35°C. Container must be fabricated and assembled in accordance with details included in CVI Corporation's drawings A458-1800 through A458-5821 on file with this Board. Except as otherwise provided herein, the container must be qualified and prepared for shipment in accordance with the Callery's design and performance specifications on file with this Board.

7. SPECIAL PACKAGING REQUIREMENTS.

- a. All diborane shipments, including those which qualify as "empty" by subparagraph (c), must comply with the following:
 - i. In addition to the flammable gas label and the poison gas label, each outside shipping container must bear a conspicuous label reading as follows: "IF NOT DELIVERED BEFORE CARRIER MUST ADVISE (Insert name and address of shipper), ALSO THE BUREAU OF EXPLOSIVES, WASHINGTON, D. C., BY WIRE." The date inserted in the blank space on this label must not be in excess of the number of days prescribed herein from the date shipment is offered for transportation.
 - ii. Container must have not less than 108 pounds of dry ice in dry ice chamber on the day shipment is made.
- b. All diborane shipments except those which qualify as "empty" by subparagraph (c) must also comply with the following:
 - i. Filling is to be by weight only.
 - ii. Containers precooled to minus 80°C. with liquid nitrogen must reach destination within 15 days from date of shipment. True temperature at or below -80°C. must be confirmed by pressure of 18 psig or lower on date of shipment.
 - iii. Containers cooled to minus 70°C. with dry ice (or liquid nitrogen) must reach destination within the following schedule.
 - (a) 15 days from date of shipment when diborane net weight is 175 to 200 pounds.
 - (b) 12 days from date of shipment when diborane net weight is 100 to 174 pounds.True temperature at or below -70°C. must be confirmed by pressure of 32 psig or lower on date of shipment.
- c. Container shipped as "empty" of diborane must also comply with the following:
 - i. Must be verified to be "empty" by one of the following:
 - (a) The empty weight must not exceed the marked tare weight by more than 5 pounds, or

- (b) Level of liquid diborane must be below the bottom of the dip tube. Loss of liquid seal will be evident by ability to vent gas pressure from the container (to user's tank or other proper vent system) through the dip tube.
- ii. Pressure should be vented to between 25 and 50 psig at the time container is emptied.
- iii. Containers shipped as empty must reach destination within 15 days from date of shipment.
- d. Provisions for shipment of methanol are as follows:
 - i. The only time methanol may be shipped is to satisfy contractor's qualification acceptance tests. Under these conditions neither the transit time restrictions nor the requirement for precooling apply.
 - ii. Additionally, the requirements of subparagraphs (a), (b), and (c) of this paragraph and the entire paragraph (9) do not apply to methanol shipments.

8. MODES OF TRANSPORTATION AUTHORIZED - Motor vehicle.

9. SPECIAL TRANSPORTATION REQUIREMENTS.

- a. A copy of this permit, kept current, must be carried aboard each motor vehicle.
- b. Shipments of diborane made under the terms of this permit must be delivered within the time prescribed in subparagraph 7(b).
- c. Each shipping paper must show thereon, following the commodity description, the notation, appropriately executed:

DOT SPECIAL PERMIT NO. 6522
DATE OF SHIPMENT _____
IF NOT DELIVERED BEFORE _____
DAYS, CARRIER MUST ADVISE
BUREAU OF EXPLOSIVES, WASHINGTON,
D.C. BY WIRE.

- d. Each shipper must require acknowledgement of receipt of shipment from consignee by wire, to be confirmed in writing, and must promptly notify the Bureau of Explosives (AAR) of any such shipment not received at destination within two days after shipment is due.

- e. Any common carrier by motor vehicle transporting diborane under the terms of this permit must be specifically approved by the Federal Highway Administration.

10. REPORTING REQUIREMENTS. Any incident involving loss of contents of the package must be reported to this Board as soon as practicable.

11. EXPIRATION DATE. September 15, 1973.

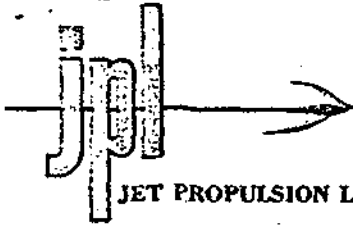
Issued at Washington, D.C.:

W. R. Fiste
For the Administrator
Federal Highway Administration

(DATE)

Address all inquiries to: Secretary, Hazardous Materials Regulations Board, U.S. Department of Transportation, Washington, D.C. 20590.
Attention: Special Permits.

Dist: a, d



JET PROPULSION LABORATORY California Institute of Technology • 4800 Oak Grove Drive, Pasadena, California 91103

September 22, 1972

Refer to: 384-WEP:lg
384CD-72-729

Office of the Secretary of
Transportation
Washington, D. C. 20590

Attention: Mr. James Grothe
Chief, Special Permits Branch
Office of Hazardous Materials

Dear Mr. Grothe:

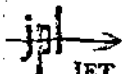
Subject: DOT Special Permit No. 6522

Special Permit No. 6522 applies to an insulated container specially designed and fabricated for the transportation of up to 200 lb_m of diborane. At present, the permit is only for quantities of from 100 to 200 lb_m of diborane, with shipment time of from 12 to 20 days, depending on the load and the initial cooldown temperature.

The Jet Propulsion Laboratory anticipates the need to make local transfers of less than 100 lb_m lots of diborane in support of various NASA contracts. These transfers can usually be made in one day, pickup to delivery. However, for conservatism, we ask that the permit be amended to allow shipment of 5 to 100 lb_m of diborane when:

- a. The container and its contents are precooled to -70°C or lower, and the cooldown temperature is confirmed by a measured ullage pressure of 32 psig or lower on the date of shipment.
- b. The container has not less than 108 lb_m of dry ice in the dry ice compartment on the day of shipment.
- c. The container reaches its destination within three days from the date of shipment.

In support of this request, I enclose a graph showing the results of a dry ice cooldown test made with the empty container exposed to the sun at our test station



Office of the Secretary of
Transportation

-2-

September 22, 1972

on the grounds of Edwards Air Force Base, in the Mojave Desert. The empty container had been cooled down and recharged several times with dry ice for about two weeks, so it was in thermal equilibrium at about -77°C when the final recharge was made on September 1, 1972. The curve shows that if the recharge had been to 108 lb_m of dry ice the period of time taken to evaporate the dry ice and increase the container temperature to -35°C would have been over seven days.

The presence of diborane in the container would probably not have increased the dry ice evaporation time, but would have increased the warmup portion of the time because of the increased heat capacity of the container plus its contents.

This test data indicates that the requested modification to Special Permit No. 6522 is conservative by a factor of more than two.

JPL anticipates having to make a transfer of approximately 90 lb_m of diborane to Rocketdyne, a distance of about 100 miles, during the week of October 10. This is a most conservative case, since the transfer will be completed in one day and the 90 lb_m of diborane will add appreciably to the heat capacity and warmup time of the shipment.

It will be appreciated if permission to make this one planned transfer can be obtained by the October 10 date, as timely performance by Rocketdyne on a NASA contract is involved.

Very truly yours,

Walter B. Powell
Member of Technical Staff
Liquid Propulsion Section

Enclosure: Graph

cc: Mr. K. W. Beahm, Callery Chemical

Test of Empty B₂H₆ Shipping Container

9-1-72 thru 9-11-72

